MPT-2 Titrator

& Degasser

user manual

Malvern

MPT-2 & Degasser User Manual

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Table of Contents

1

Introduction and hardware

Introduction

This manual details the important features of the Zetasizer Nano accessories; the MPT-2 Multi-purpose titrator and Vacuum degasser.

Accessory	Part number
MPT-2 Multi-purpose titrator	ZEN1001
Malvern Vacuum degasser: Auto-degas unit for the MPT-2	DEG0003

This manual is a supplement to the following manuals:

- **Zetasizer Nano User Manual**
- **Zetasizer Nano Basic Guide**
- **Zetasizer Nano Accessories Guide**

Warning!
The accessories or the samples to be measured may be hazardous if misused. Users **must** read the **Health and Safety** information in the **Zetasizer Nano Basic Guide** before operating the system.

This manual focuses on specific issues of the Zetasizer nano accessories that are not covered by the above manuals. Within the following chapters the manual will detail:

Introduction and Hardware

This chapter serves as the introduction to the accessories and describes what the accessories are and explains in simple terms how they work. It also identifies the physical features of each accessory and how to connect each one to the Zetasizer Nano optical unit.

Software and Control

This chapter describes the accessory software controls identified in the Zetasizer Nano user manual and explains how to use the accessories to make measurements on the system.

Maintenance

This chapter covers all the user maintenance procedures for the accessories. This includes a maintenance schedule and associated maintenance procedures for inspecting and cleaning each dispersion unit and its respective components.

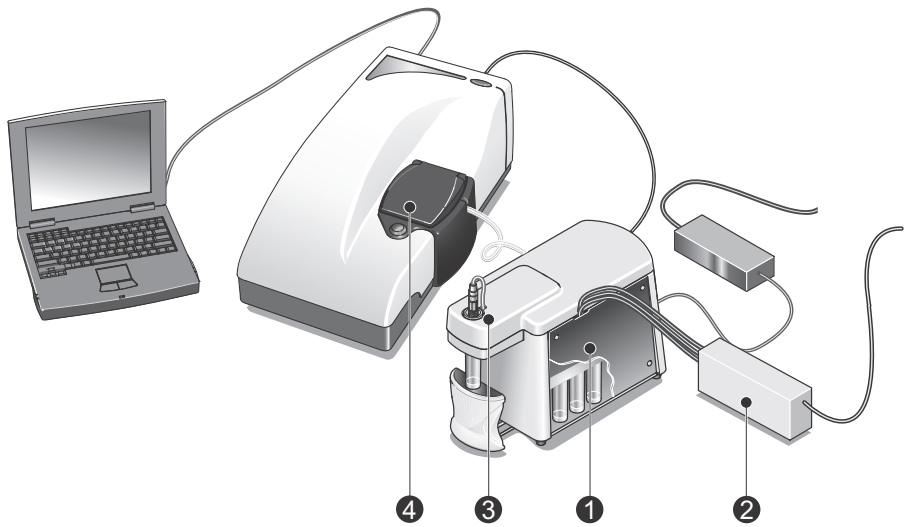
Appendices

This chapter / appendix details the useful specifications of each accessory and identifies the chemical compatibility of the accessory components that may come into contact with the sample. Additionally it provides the important regulatory information to which the accessories are compliant.

MPT-2 Multi-purpose titrator / Vacuum degasser - Overview

What does the Titrator do?

The principal function of the Titrator is to allow zeta potential, size or intensity measurements to be made, while adding quantities of additives (titrants) to the sample when performing a particular titration type, i.e. pH, Dilution or Additive. This allows the effect of adding these titrants on the measurement to be investigated.



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The heart of the titrator ① is the dispenser unit. The sample to be measured is placed in a disposable sample container. The dispenser unit has an internal pump that circulates the sample through the cell in the optical unit. (A size flow cell will be required if size measurements are to be made).

The dispenser unit has three titrant containers that can be filled with either an acid, base or some other additive. The titrants used can be different concentrations of the same acid or base. This will improve the accuracy of obtaining a target pH or concentration and limit the quantity of titrant used.

The syringe system can deliver a precise quantity of titrant from one of the titrant containers into the sample container.

Additionally a Vacuum degasser ②, placed between the titrant containers and the sample container, removes dissolved gasses from the titrants used.

The software can be set up so that measurements are made under a number of sample conditions. The sample is stirred, using the stirrer attachment ④, pumped through the flow cell and then measured at each of these conditions. In this way the effects of adding various amounts of these titrants on the zeta potential, size or intensity can be investigated. This method is known as a titration measurement.

A pH probe ③ continually monitors the pH of the sample, while a temperature controller in the optical unit maintains the sample at a selected temperature.

A pinch valve ④ integrated into the Zetasizer Nano basin moulding pinches the tubing and ensures that fluid is stationary during measurements.

Once the measurement sequence is complete, the sample and titrants can be flushed from the system. The sample and titrant containers, if required, are replaced with ones containing flush fluid. The fluid is then circulated through the system to a waste container.

Vacuum degasser

The Vacuum degasser is a high-efficiency in-line module that removes dissolved gasses from solvents used in flowing systems, and more specifically from the titrants used in the MPT-2 Multi-purpose titrator. Its design assures reliable continuous operation and the highest level of continuous performance available without the need for helium degassing. Up to 3 titrant lines may be degassed simultaneously by one unit.

Internally, the titrant flows through a short length of **Teflon AF®** tubing which is located in a vacuum chamber. Within this chamber a partial vacuum is maintained by a constantly running, low RPM (revolutions per minute) vacuum pump.

- Dissolved gasses migrate across the tubing wall under a concentration gradient produced by the vacuum as the titrant flows within the coil.
- Gasses removed are expelled, and the chamber is maintained at a constant, preset vacuum level by varying the vacuum pump speed as needed.

An additional port in the vacuum pump continually flushes the pump head with a small "bleed" of air to remove any titrant vapours which may enter the pump from the vacuum chamber. This air bleed eliminates the need for any solenoid valves within the system. This patented design results in zero vacuum "hysteresis".

Control of the Titrator

The MPT-2 titrator can be controlled by two methods - both via the Zetasizer Nano application software:

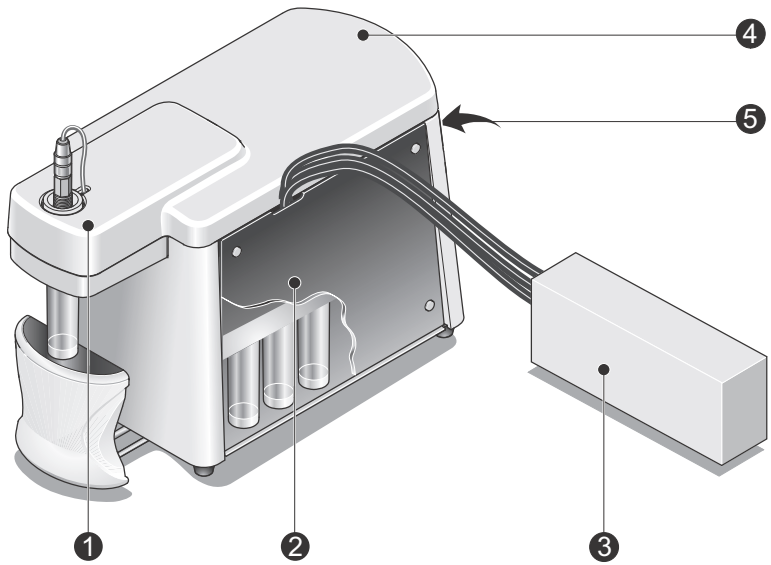
- Automatically, as part of measurement protocol when controlled through a Standard Operating Procedure (SOP). The software tells the user what they need to do as the measurement progresses.

- Alternatively the accessory can be controlled by a separate manual accessory control dialogue. This will enable simple control of the dispersion unit, allowing individual selection and operation of the units functionality. This is useful for performing evaluation methodology before constructing a SOP.

  Additionally the manual accessory control dialogue is used for checking the operation of the pH probe and for performing maintenance operations such as pH calibration. This dialogue is described later in the manual.

Features of the MPT-2 titrator accessory

This section identifies the main features of the MPT-2 titrator accessory.



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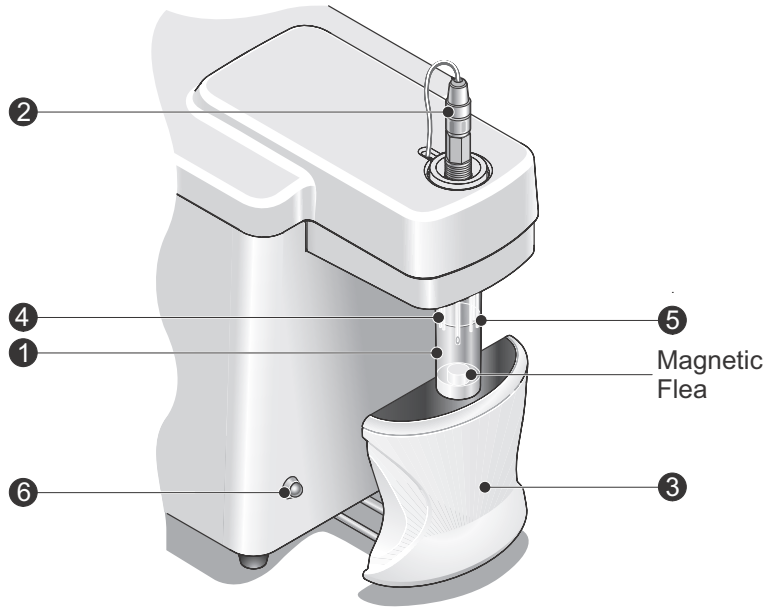
① Dispersing head	④ Dispersing area cover and tubing connection bracket
② Dispersing area	⑤ Rear Panel
③ Vacuum degasser	

Dispersion head

The Dispersion head can be thought of as a mixing bowl. The sample to be measured is placed in a container that is screwed to the underside of the dispersion head. Titrants are added and the sample stirred. The dispersion head also houses the pH probe.

The illustration below identifies the main features of the dispersion head.



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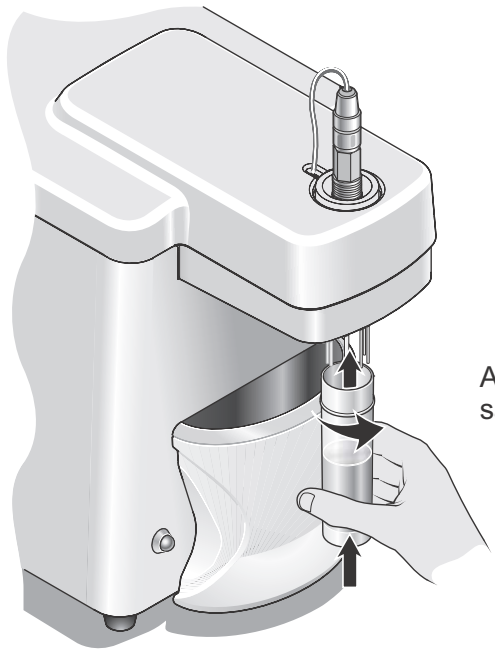
① Sample container

The sample container is the mixing and reaction vessel for the measurement.



Attaching the
sample container

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The sample container initially just contains the sample to be measured. Titrants are added to the sample through the titrant tubes and the dispersion is stirred.

Two sizes of containers can be used; the 'standard' container as shown above, and a 'large volume' container, which will generally be used for dilution titrations.

To attach and remove the 'standard' sample container, simply screw the container into, or unscrew the container from, the dispersion unit; as shown above.

Instructions on fitting the large volume container are described in the **Titrants SOP** dialogue section in the next chapter.

Be careful not to splash any sample when inserting and removing the sample container.

The sample containers are disposable - replacement containers can be obtained directly from Malvern instruments.

Note
This manual will assume that a standard sample container will always be used, unless otherwise stated.

② pH probe

The pH probe measures the pH of the sample. The performance of the pH probe will deteriorate over time, though correct maintenance will extend its life and ensure rapid and accurate titrations. It is recommended that the probe is replaced at least once a year.

Caution!
The probe must never be allowed to dry out. If it does it may need replacing. See **Chapter 5** for details on maintaining the probe.

③ Magnetic Stirrer

The stirrer agitates the sample and aids titrant dispersion.

The stirrer motor is pulled forward from the front of the titrator to sit directly below the sample container. A **magnetic stirrer 'flea'** placed in the sample container will agitate the sample once the stirrer is turned on during a titration or pH calibration.

The speed of the stirrer is set through the **Titrants** SOP dialogue. The optimum speed will depend upon the type of sample being titrated. Care should be taken when adjusting the stirrer speed as air may be introduced into the sample. If in doubt, set the stir speed check box to **Automatic**.

④ Sample exit and return tubes

The sample is pumped to and from the cell through these tubes. The ends of the sample exit and return tubes must always be below the surface of the sample, for two reasons:

1. If the sample exit tube is above the level of the sample, no sample can be pumped and circulated.
2. Bubbles may be generated from the dropping of sample back into the sample container.

⑤ Titrant tubes

The measured dose of titrant from the titrant containers enters the sample container through these tubes. The ends of the titrant tubes must always be below the surface of the sample, for two reasons:

1. If small amounts of titrant are dispensed these may stay attached to the end of the titrant tube. Immersing the tubes in the sample will break the surface tension.
2. Bubbles may be generated from the dropping of titrant into the sample.

The titrant tubes are the same as those used for the sample. If required the titrant tubes can be extended to use a larger volume titrant container. A cut-out is provided at the top of the dispersion area cover for the exit of the tubes.

Note

For protein measurements where only a minimum sample volume is desired, loosen the tubing clamp and carefully pull both **sample** and **titrant** tubing down towards the bottom of the container - see the **Tubing connection bracket** section later in this chapter. Ideally the stirrer is not used. It is not required with protein measurements when the minimum volume is required.

⑥ Status indicator

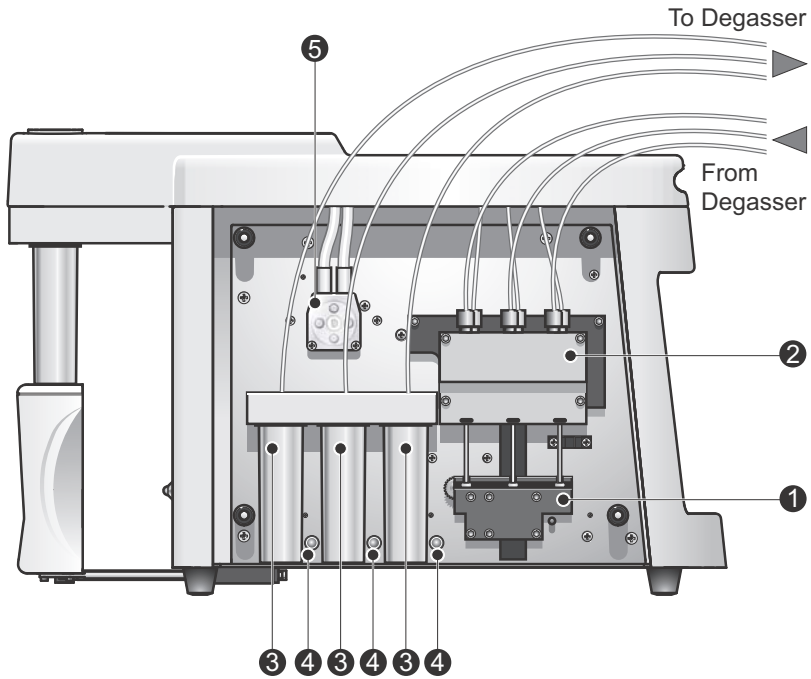
This indicator shows the status of the titrator. The indicator will show:

- **Red** if the titrator has encountered an error condition.
- **Green** if the titrator is in operation.
- **Amber** if the titrator is in standby mode.

Dispersing area

Warning!
For safety reasons the dispensing area cover must always be in place during a measurement sequence.

The dispensing area houses the dispensing syringes, titrant bottles, valves and the pump head. The main features of the dispersing area are described below.



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① Dispensing syringes and syringe drive

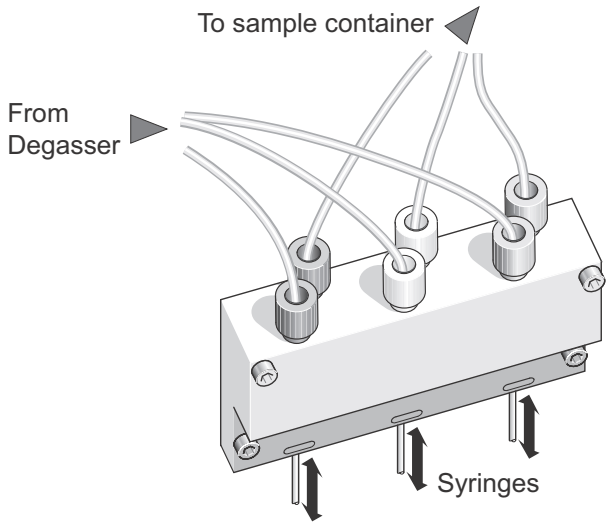
The syringes are the mechanism for dispensing precise amounts of titrants from the titrant containers to the sample container. When a titrant is requested the syringe drive will descend to first obtain the titrant from the container, and then ascend to deliver the titrant to the sample container; this is done in conjunction with the manifold assembly and solenoid valves described below.

② Manifold assembly and solenoid valves

The manifold assembly and solenoid valves control the flow of sample through the cell. These valves are either opened or closed depending on the requirements of the prime and titration sequences.

The solenoid valves open and close to control the flow of the titrant from the container, through the manifold assembly, to the sample container.

If only one titrant is requested to dispense, its respective solenoid valve will open to allow the titrant to be delivered when the syringe ascends. The other two solenoid valves will stay closed so any titrant dispensed will return to the correct titrant container.



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③ Titrant containers

The titrant containers contain the solutions that will be added to the sample during a titration measurement sequence. Each container can hold either acid, base, salt, or an additive. The tubing connectors from the top of the titrant containers are colour coded at the manifold to match LED indicators (below) and the colours in the **Titrants** SOP dialogue.

How to fill, change and insert the titrant containers is described in **Chapter 4**.

④ Titrant indicators

Each titrant container has an LED indicator alongside that illuminates whenever that titrant is being dispensed. The three LEDs are colour coded (red, yellow and green) to reflect the colour coding in the **Titrants** SOP dialogue.

⑤ Pump head

The pump head is used to pump the sample (or cleaning fluid) through the cell. The speed of the pump is set through the **Titrants** dialogue.

Vacuum degasser

The vacuum degasser removes any dissolved gasses from the solvents used in the titrants containers.

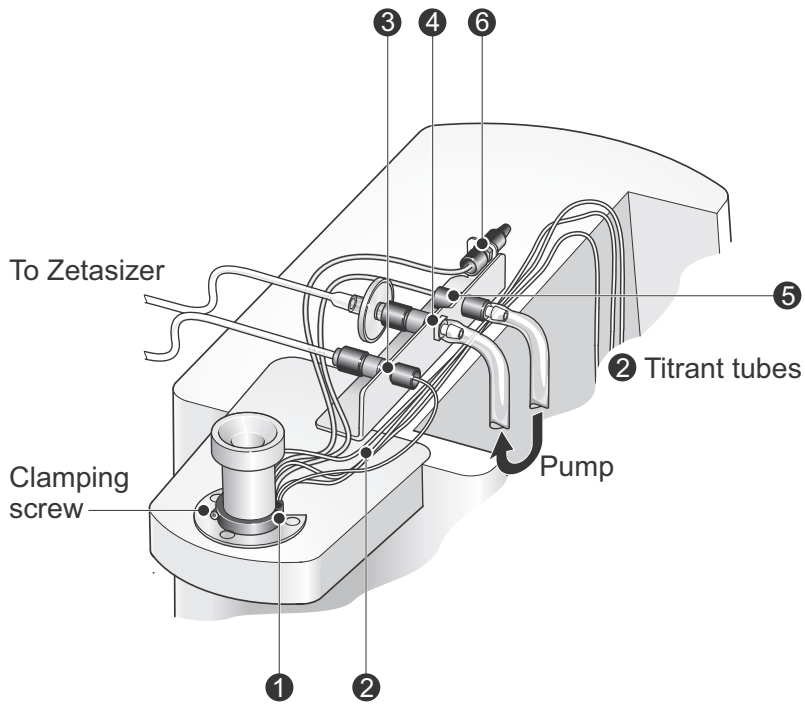
The features of the degasser are described later in this chapter.

Dispensing area cover and Tubing connection bracket

The dispensing area has a removable cover that allows access to both the titrant and sample tubing, and the sample filter. The cover simply pulls up and off the unit.

All the titrator tubing is routed around the tubing connection bracket underneath the dispenser lid. The illustration below shows details the various connections. How to connect or disconnect the tubing is described in the **Chapter 5**.

To access the bracket simply pull the dispersing area cover off.



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① Tubing entry and tubing clamping ring

All tubing enters the sample container through entry holes in the container holder. The tubing includes three titrant tubes, purge tube, sample in and sample out tubing. These are described in the following paragraphs.

Adjusting the tubing length

Depending upon the titration sample volume used and the type of titration being performed, the length of the tubes can be altered to be either higher or lower within the sample container. This is done as follows:

- Loosen the **clamping screw**, to loosen the tubing **clamping ring**.
- Carefully pull the tubing either up or down to the required height.
- Tighten the **clamping screw** to secure the tubing to be clamped in position.

Note
The clamping ring can be used for securing both thin and thick tubing diameters in place. Thick tubing (1/8") is supplied as standard for connecting the sample flow in and out of the sample container.

② **Titrant tubes**
The titrant tubes are fed directly from the manifold to the sample container.

③ **Sample in connector (from Zetasizer)**
The sample that has been measured is re-circulated to the sample container via this connector.

④ **Sample out connector (to Zetasizer)**
The sample to be measured is recirculated through this connector to the cell in the optical unit. For biological samples, a syringe filter of minimum size 0.22μm can be placed on the exit of this connection to filter oversized particles. This filter should be replaced regularly to maintain optimum filtration.

⑤ **Sample to pump**
This connection feeds the sample from the sample container to the pump before passing to the sample out connector.

⑥ **Purge connector**

Warning!
If a Nitrogen supply is used the system must be located in a well ventilated environment. Turn **off** the supply when not in use.
See Appendix B for a requirement specification for the Nitrogen supply.

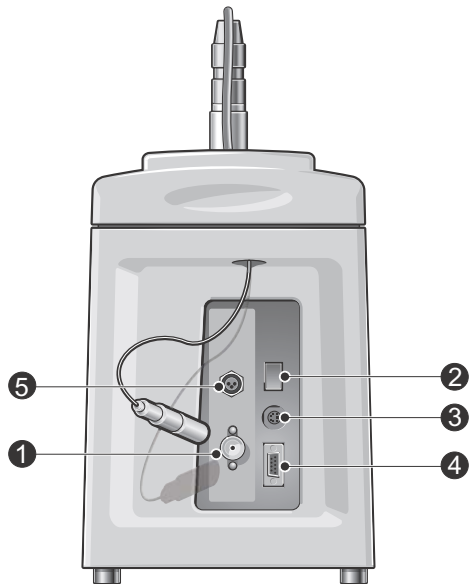
The purge connection is provided to enable connection of a Nitrogen purge supply. This is used to prevent any absorption of oxygen that may change the pH characteristics of the sample, i.e. cause a pH drift. Though the amount of pH drift is dependent upon the alkalinity of the sample, even for a sample with a low alkalinity this drift will be noticeable. The need to eliminate this drift is therefore of particu-

lar importance when performing protein charge titrations; pH drift during this operation will lead to extended titration times.

The amount of Nitrogen needed to prevent oxygen absorption should be just enough to cover the immediate space above the sample. The Nitrogen can be introduced into the container by blanketing the area directly above the sample or by bubbling the nitrogen through the sample. If too much nitrogen is introduced, or the flow rate is too high, there is a risk of bubbles being generated in the sample.

Rear panel

The rear panel contains all electrical connections to the computer and optical unit.



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① pH probe connection

The pH probe is connected to the pH probe connection on the rear of the titrator.

The pH probe connectors must be clean and dry at all times. Contaminated connectors may affect the performance of the probe.

② On/off switch

The power on/off switch for the titrator.

③ Power input

Power input socket for the titrator. The external power supply is plugged here.

④ Communications connector

Connects to the RS232 port on the Zetasizer Nano series instruments to allow the software to control the titrator.

⑤ Stirrer connector

Connection for the magnetic stirrer.

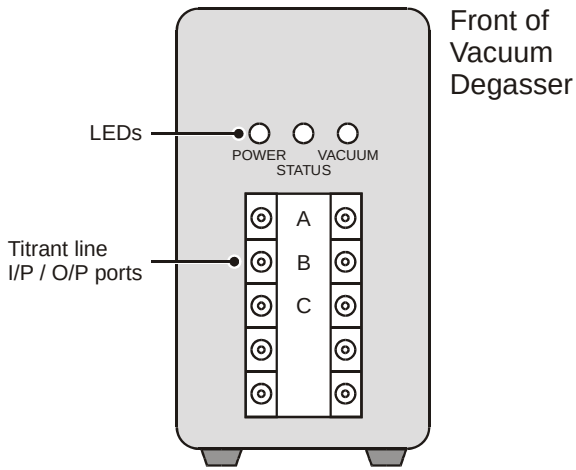
pH probe

The pH probe connects to the rear panel of the titrator.

Features of the Vacuum degasser accessory

This section identifies the main features of the Vacuum degasser accessory.

Front Panel Connections



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Titrant line ports

There are 3 degassing channels labelled A to C on the front of the degasser. Each channel consists of a pair of female connectors which provide the input and output ports for running the titrant lines through the vacuum degasser to the titrator.

Note
Flow direction is not critical.

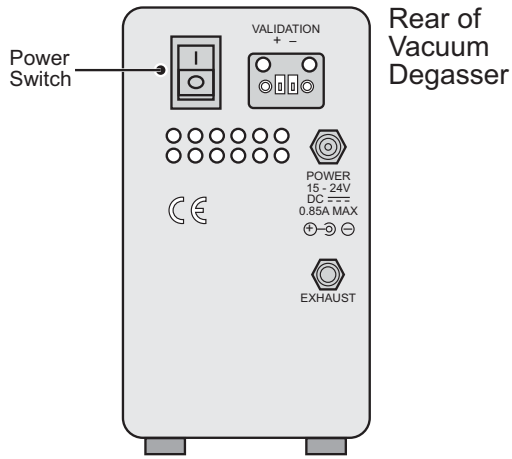
Plugs are provided to seal the ports of any unused channels. This must be done to ensure the Degasser works correctly.

Front Panel Indicators

Three LEDs are located on the front of the instrument above the titrant inlets and outlets; these indicate the following.

Power (Green)	Indicates when power is applied to the Vacuum Degasser - cable is plugged in and Power switch turned ON.
Status (Yellow)	Indicates when vacuum level is outside the acceptable operating range. This LED will light at initial power-up and remain on during pump-down. It will turn off after a few minutes when the vacuum level goes below 100 mm of Hg absolute. If an error condition occurs, this LED will flash in one of two modes: Flashing on and off in even 1-second intervals: pump was not able to reach vacuum set point, indicating a possible leak in the system. Flashing on for 1 second and off for 2 seconds indicates a vacuum signal error.
Vacuum (Green)	Indicates when vacuum level is within the acceptable operating range. The LED will light up after the initial pump-down, and remain on as long as the Vacuum Degasser is powered up and the vacuum level is below 100 mm of Hg absolute.

Rear Panel Connections and Controls



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Power Connector

Caution!
Only use the AC power adaptor as supplied from Malvern Instruments.

Connect the output plug from the AC power adaptor to this connector.

See **Specifications** chapter for further power requirement information.

Power Switch

The On/Off power switch turns the Vacuum Degasser On,

O = Off
I = On.

Exhaust Port

Any gas pumped out of the vacuum chamber exits the unit through the exhaust port.

Validation Connector

Depending on the model, there may be a 2-pin receptacle labelled "Validation" located next to the power switch. This receptacle and its associated screw-lock plug allow a validation signal from the Vacuum Degasser's control circuit to be sent to a computer or data system. This validation output indicates vacuum level (see **Specifications** section for details).

2

Connection and accessory preparation

Introduction

This chapter describes the actions necessary for preparing the titrator and degasser accessories ready for measurements to be performed. It covers:

- Connecting the MPT-2 Titrator and Vacuum degasser to the Zetasizer Nano.
- Preparing the titrant and sample tubing and containers.
- Calibration of the pH probe.

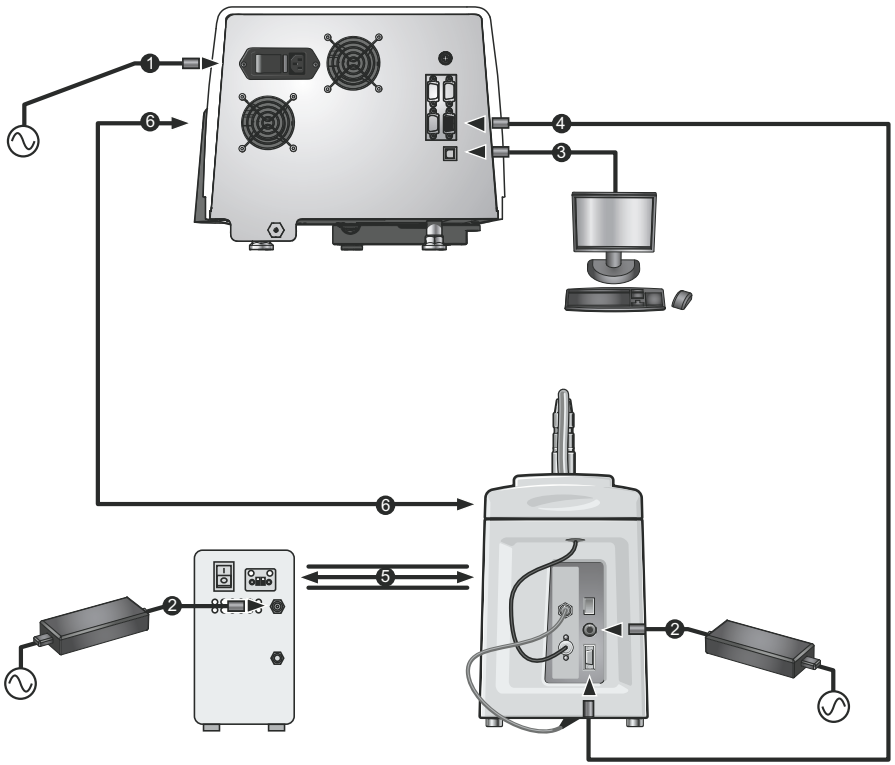
Connecting the titrator and degasser

This section gives instructions on how to connect the accessories to the Zetasizer Nano optical unit. Initially the accessory will have been commissioned by a Malvern Instruments representative.

Note that when the accessory is to be moved short distances, then a sample container containing water should be attached to the dispersion head to prevent the pH probe from drying out. If the accessory is to be moved greater distances then the pH probe should be removed and its protective cover attached to prevent the probe from drying out.

Follow the installation instructions for the Zetasizer Nano as detailed in the Zetasizer Nano user manual, then connect the Titrator as described below.

Connections



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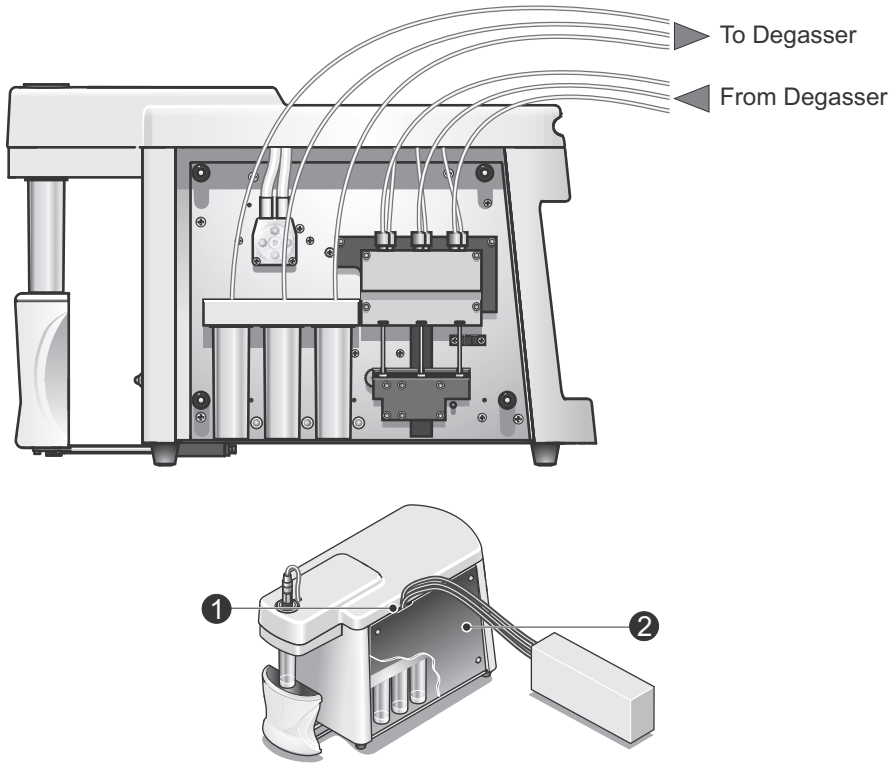
① Power cable (direct)	④ RS232 connection to MPT-2 Titrator
② Power cable from external PSU	⑤ Titrant tubing to / from Degasser
③ Computer connection (USB)	⑥ Sample tubing to / from Nano cell

Connecting the titrant tubing between the titrator and degasser

Titrant lines to be degassed are connected to the degasser's front panel ports, as detailed below. Any **unused ports** must be **plugged** to enable the degasser to operate correctly.

The titrant tubes are fed from the titrant containers through the space ① at the top of the dispensing area cover towards the degasser input ports, and then back again towards the manifold assembly ②.

Note
Initially the accessory will have been commissioned by a Malvern Instruments representative. The below information describes how to fit any new tubing.



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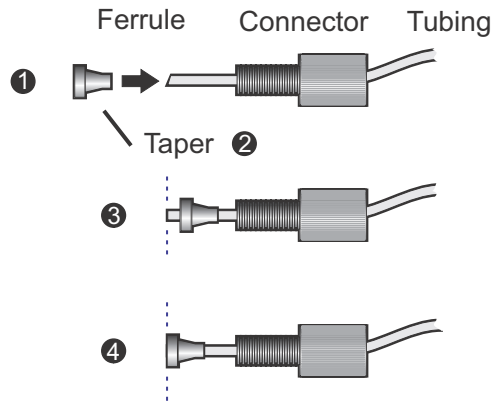
Connecting the tubing (titrator to degasser)

- Position the titrator and degasser suitably for operation, and ensure the tubing lengths will comfortably reach the degasser from the titrator.
- Push a line of tubing through the titrant container clamp above the titrant containers until it reaches the base of the container. Run the line of **tubing** from the titrant containers in the titrator to the degasser input.
- Push the tubing through the **connector** and slide a **ferrule** over the tubing end ①; note the direction of the ferrule **taper** ②.



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- Cut the **tubing** so the end is flat ③.
- Move the tubing so its end is **flush** with base of the ferrule ④.
- Screw the connector into one port on the front of the degasser (Channel A, for example). The direction of flow through the degasser is not critical.

Note
Plastic connectors should only be tightened by hand. Over-tightening them will damage the threads.

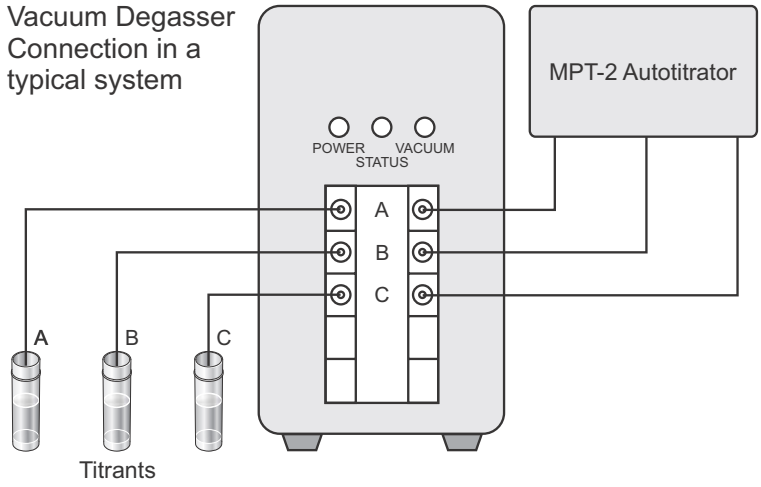
- Repeat the steps above to connect additional titrant lines to the degasser.

Connecting the tubing (degasser to titrator)

Caution!
Never connect the Vacuum Degasser to the output side of the titrator pump. The high pressure may cause permanent damage to the degassing membrane.

- Run the line of **tubing** from the titrant containers in the titrator to the degasser input.
- Follow the instructions above on how to fit the **connector** and **ferrule** to the tubing end.
- Connect the completed tubing assembly to the degasser and the titrator manifold input.
- It is recommended that tubing is connected to the manifold in the same order as they exit the titrant containers.

The following illustration shows how the finished titrant connections should look. Note that the direction of flow through the degasser is not critical.



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Once all required titrant lines have been connected to the degasser, plug all unused ports with the spare plugs supplied. Press these in by hand.

Connecting the Titrator to the Zetasizer Nano

Follow the guidelines on inserting the cell in the Zetasizer Nano manual.

The tubing has to be attached correctly between the cell used and the titrator unit itself. How the tubing is connected to the cell is dependent upon the cell and the measurement being performed.

Connecting from the Titrator to the Nano

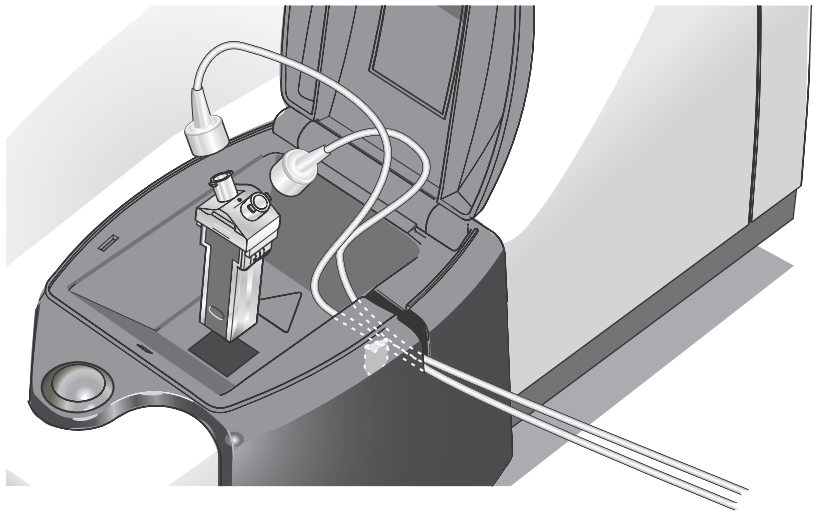
Follow the instructions below for connecting the cell. For each cell, minimise the tubing within the cell area before inserting into the pinch valve channel.

Zeta potential measurements

The tubing is attached to the folded capillary cell (DTS1070/DTS1061) using 'Luer lock' connectors.

With a half-turn these secure to the Luer fittings on the top of the cell (do not over-tighten). The tubing is then inserted into the pinch valve channel; push **both** tubes down into the pinch valve on the side of the cell area.

If a **flowcell** is used, insert the sample tubes into the threaded inserts and screw into the top of the flowcell, and then push **both** tubes down into the pinch valve on the side of the cell area.



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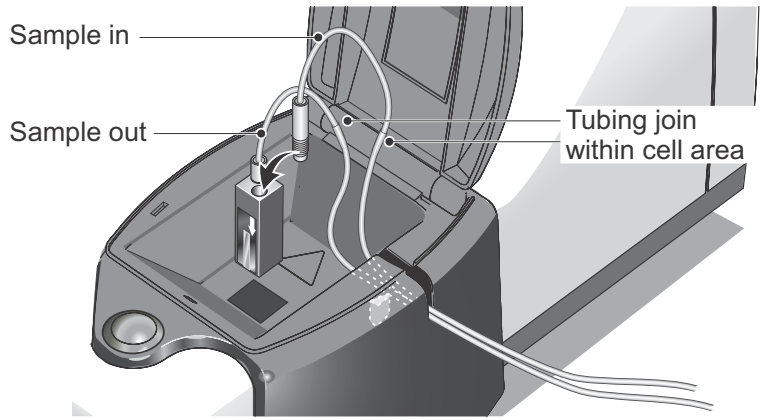
Note

The pinch valve manufacturer recommends that a vegetable-based oil (e.g. Castor oil) is used to lubricate the section of tube that is inserted into the pinch valve. This is done to help minimise friction, though testing by Malvern Instructments has not shown this to be essential.

Size measurements

The tubing is attached to the sizing flowcell (ZEN0023) using threaded inserts. As with the folded capillary cell above, push **both** tubes down into the pinch valve on the side of the cell area. Ensure the join between the PTFE and silicone tubing is within the cell area.



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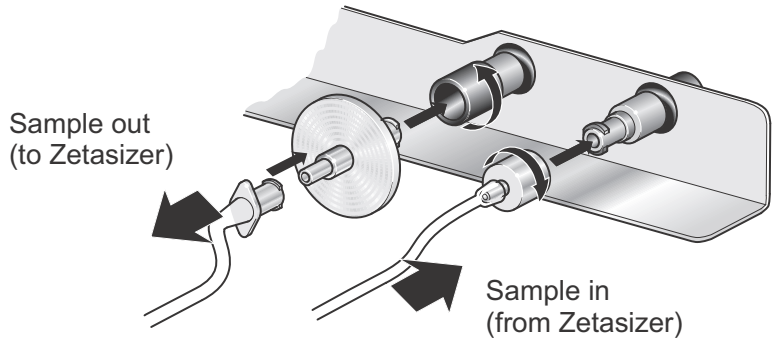
Connecting from the Nano to the Titrator

The connections used on the titrator are of the "Luer lock" variety. The diagram below shows how to connect the tubing, from the cells mentioned above, to the tubing connection bracket. Similar connections are used throughout the unit; the connections may differ slightly, but the connection principle is the same.



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If a filter is used, attach this to the sample out (To Zetasizer) connection in the same manner as the direct tubing, then connect the tubing to it.

Preparation for measurement

Some setup procedures will have to be performed daily, while others will only have to be performed at much longer intervals.

Typically, setup before performing a measurement will involve one or more of the following operations:

- **Filling the titrant containers.** Only required when the containers are nearly empty.
- **Priming the titrant syringe pumps and tubes**. Usually done at the beginning of the day or at the start of a new measurement session.
- **Calibrating the pH probe.** Usually performed before each titration session.
- **Preparing the measurement sample**. Performed before each titration session.
- **Filling of the measurement cell.** Performed before each titration session.

To perform these operations the titrator manual control dialogue will be required. Where necessary refer to the Software and control chapter.

Caution!
The pH probe will require special care. Never allow the pH probe to dry out. It is important to read the section on the care of the pH probe in **Chapter 5**.

Filling and changing the titrant containers

The three titrant containers are typically filled with acid, base, salt, or other additives. For most applications all that is required is to check that there is enough titrant in the containers and re-fill if necessary.

Alternatively, if different titrants are continually being used, one of the titrant containers may need to be changed prior to the measurement.

Always consult the Materials Safety Data Sheets for any titrant being used for information on safe handling.

Changing and Re-filling the containers

The titrant containers will hold up to 25ml of titrant. When the level reaches approximately 5ml, it is recommended to re-fill the container. To refill:

- Remove the titrant container by lifting the container cover up and sliding out the container.
- Dispose of the remainder of the titrant and rinse the container.
- Prepare 25ml of titrant to the correct concentration (see **A note on titrant concentration** later in this section).
- Add the titrant to the container.
- Re-fit the container. Lift the container cover up and slide the container in to the empty holder position. Close the cover ensuring all containers are fitted correctly into the cover.
- Prime the titrant tubing using the **Manual control** dialogue, as described below.

Note
If changing the titrant type used, it is important to clean the tubing and containers if re-using. Refer to the **Maintenance** chapter for details. Also change the titrant settings in the SOP as appropriate for the new titrant, i.e. name, type, concentration.

A note on titrant concentration

Preparing and specifying the concentration of the titrants is critical to achieving accurate results. The concentration specified within the **Titrants** SOP dialogue must be accurate to within 10%.

A concentration of 0.25M is satisfactory for most measurements. In some situations it may be wise to vary this. For example, if a large number of points is to be used in a titration measurement, or if performing pH measurements close to pH 7, a lower concentration may be needed.

The titrator can accurately dispense small volumes so concentrations of titrant up to 1M can be used.

It is recommended that pre-diluted titrants are purchased. Either use titrants that are diluted to the exact concentration required, or purchase titrants of a known Molar concentration that can be easily diluted further.

For titrations that cover a wide pH range it is useful to use two concentrations of titrant, e.g. 0.25M of acid and 0.01M of the same acid. This will ensure that the pH values achieved are closer to the requested values.

Priming

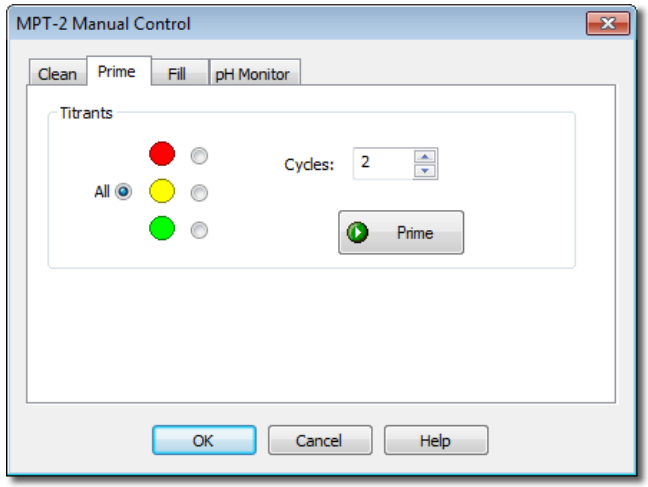
Caution!
DO NOT prime the membranes by pushing titrant through the degassing systems. This technique can generate several hundred pounds of pressure which might rupture the Teflon AF® membrane. The maximum recommended pressure on the membrane is **0.7 MPa** (**100 psig, 7 Bar**).

Priming is the process of ensuring that all the titrant delivery tubes are free of air and full of liquid. Generally this is done after changing titrants or after the system has not been used for a time, e.g. overnight.

Over time, the titrant may evaporate from the end of the titrant tube and therefore it will not deliver the correct volume when first used. It is recommended that the dispenser unit is primed every morning or at the beginning of a new session. Select one prime cycle when performing a daily prime.

The dispenser unit should also be primed when a titrant container is changed in order to remove any traces of the original titrant that may be left in the titrant tubes. Three prime cycles will be sufficient to remove previous titrant.

Each titrant delivery tube can be individually primed, or **all** tubes can be primed simultaneously. The coloured indicators on the dialogue match the LED indicators next to the titrant container, and also the titrant tubing connectors.



Priming the titrant tubes:

- Place the filled titrant containers under the container cover, and under the titrant tubes that are to be primed.
- Attach a sample container, containing a small amount of clean water, to the dispersion head; this is used to catch the titrants during the priming process. Water in the container is not essential, but will eliminate the effect of surface tension at the end of the titrant tube.
- Open the titrator application by selecting the **Manual control** icon.
- Select the radio button for the titrant tube that is to be primed, or **All** to prime all three titrant delivery tubes at once.
- Select the **cycles** required and press the **Prime** button to begin the operation. Each cycle will pump the volume of the titrant tubes from the container. (Note that, once started, pressing **Prime** a second time will stop the operation).
- Priming will stop once the number of cycles has been completed.
- When the prime operation has finished, titrant should be visible in the bottom of the beaker. If none is dispensed, the **Prime** button should be pressed again to repeat the process.

Note
The titrant tubes can also be cleaned using the same tab. Simply insert containers of cleaning fluid in place of the titrant containers.

Calibrating the pH probe.

The performance of the titrator relies on the pH probe being correctly calibrated. If the pH probe has not been calibrated then the pH values will be incorrect.

It is recommended that the pH probe is calibrated before each titration session.

To calibrate the pH probe the follow the instructions in the **Software** and **Maintenance** chapters.

Preparing the sample in the container

Before starting the measurement, it is necessary to prepare the sample and attach the container to the dispersion head

- First prepare the sample to be measured and place a known volume into a sample container. An initial sample volume of 10ml is recommended (5ml minimum to 25ml maximum). The preparation of the sample is of prime importance. Read the sections on sample preparation in the Zetasizer Nano manual.

Note
The sample used should be of a suitable concentration that gives an intensity of scattering within recommended values. This is checked at the beginning of each titration. If the concentration is too high the titration may proceed slowly due to the sample buffering the pH. If a message occurs indicating the sample concentration is higher than recommended, dilute the sample and restart the titration.

- Attach the sample container to the dispersion head as described in the **Introduction and Hardware** chapter.

Filling the cell

With the cell tubing connected, and the sample container prepared and mounted onto the dispersion head, all that is left to do before configuring the SOP and starting the measurement is to fill the cell.

The cell must be filled so that no bubbles are left within the sample path; this is especially important for Zeta potential measurements. Follow the instructions below:

- Open the **Manual control** dialogue by selecting **Tools-Instrument- MPT-2 Titrator- Manual control** within the Zetasizer Nano software, or by selecting the **Manual control**  icon.

- Select the **Fill** tab.
- Select the **cycles** required and press the **Fill** button(s) to begin the operation. Each cycle will pump the volume of the sample tubing and cell.
- The pump will circulate the sample through the sample tubing and cell until the number of cycles has been completed or the **Fill** button is pressed a second time. Use the pump and stirrer controls to adjust the pump and stirrer speeds during the filling - see the note below.
- Before starting the titration check that this procedure has eliminated all bubbles from the cell.

Note
Filling the cell should be performed at a lower speed than during a normal titration i.e. 40% of normal speed. This to prevent the introduction of bubbles into the tubing which will affect the measurement result, especially when performing a Zeta measurement. If bubbles are present, invert the cell while still pumping and tap it lightly to dislodge them.

Preparation summary

In summary the following operations should have been done before continuing and making a measurement.

- Install and connect the titrator and degasser, as described.
- Verify that plugs are installed in any unused ports.
- Select and fill each titrant container with the titrant(s) used for the analysis.
- Prime the titrant tubes:
- Calibrate the pH probe.
- Prepare the sample in the container.
- Fill the cell used in the Zetasizer Nano.
- **Allow the system to equilibrate for 5-10 minutes.**

Note
Always shutdown the instrument when not performing any degassing. Refer to the Maintenance chapter for instructions.

3

Software and Control

Introduction

This chapter describes the features of the software which are specific to the dispersion unit. It covers:

- Making a measurement – the basics of making a measurement using the dispersion unit using a Standard Operating Procedure (SOP).
- Manually controlling the dispersion unit. A manual accessory control dialogue is available for checking the operation of the pH probe and for performing maintenance operations such as pH calibration. This dialogue is described at the end of this chapter.

Making a measurement

Making a measurement using the Zetasizer Nano instrument is fully documented in the **Zetasizer Nano User Manuals**. Refer to that manual for details.

The MPT-2 Multi-purpose titrator can be controlled by two methods - both via the Zetasizer Nano application software:

- Automatically, as part of measurement protocol when controlled through a Standard Operating Procedure (SOP). The software tells the user what they need to do as the measurement progresses.
- Alternatively the accessory can be controlled by manually. This will enable simple control of the dispersion unit, allowing individual selection and operation of the units functionality. This is useful for performing evaluation methodology before constructing a SOP.

These are described in the following pages.

Control of the accessory via an SOP

An SOP can be configured to control all settings for the accessories automatically.

A titration measurement follows the same SOP format as performed when doing a normal Size or Zeta measurement, with a few exceptions. When a titration measurement is chosen two extra dialogues - **Titrants** and **Titration sequence** - will be included in the SOP selections.

The **SOP Editor** and setup is described in full in the **Zetasizer Nano User Manual**. Most of the SOP sections are common to Measurement types, and these are described in the above manual. The other SOP sections are specific to the Titrator accessory being used; these are described below. Also note that some of the other dialogue pages will alter slightly to accommodate extra parameters necessary to perform the titration measurement.

Creating or editing an SOP - Measurement Type selection

- To create a new SOP, select **File-New SOP**. This will open up the SOP Editor. The SOP Editor consists of several dialogues that can be stepped through by using the **Next** arrow button.
  (To edit an existing SOP, choose **Open-SOP** instead.)
- Complete the SOP Editor as described in the Zetasizer Nano User Manual.
- Once the SOP has been created, press **Finish** and save the new SOP.

The various SOP dialogues are described below.

Measurement type options

Select an **Titration** measurement and then choose whether a pH, Dilution or Additive titration is required. Lastly select the type of measurement that will be performed (Size, Zeta potential, Intensity or a combination of two measurement types i.e. Size and Intensity).

The **Measurement type** options are:

Titration type	
pH	This is a standard pH titration used to monitor solution properties, or sample size, as a function of sample pH. During a Zeta potential pH titration the Isoelectric point can be determined.
Diluton	The Dilution titration is used to monitor either sample Size or Zeta potential parameters as a function of sample concentration.
Additive	This titration is used monitor the effects of formulation additives on the Size and Zeta potential of the sample. Results are shown as a function of concentration.
Additive Log (Conductivity)	This titration is used to monitor the conductivity of a sample by adding salts. Results are shown as a function of conductivity

Sample - General options

This dialogue includes information from **both** the Size or Zeta **Sample - General options** dialogues. See description in the Zetasizer Nano manual for details. Only the **Sample viscosity option** will be shown if an **Intensity only** measurement option is chosen.

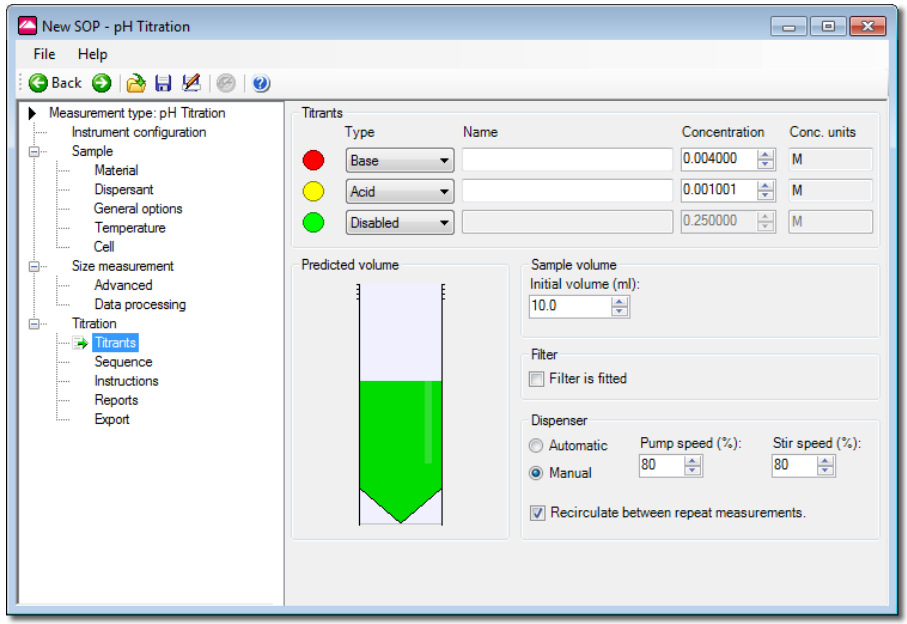
Sample - Temperature

See the description in the Zetasizer Nano manual.

Note
As the temperature probe is not temperature compensated, it is recommend that titrations are performed with ambient temperature conditions. Adjust the temperature in the SOP to the ambient value.

Titration - Titrants

This dialogue enables the titrants used to be specified, and the pump and stirrer speed to be set.



Settings	Description
Titrants	The **Titrants** Configuration area is used to set up the features of the titrants used, such as their type, name and concentration. **Type of titrant** - Specifies the type of titrant in each of the containers. Use the drop down list to select the type of titrant used, i.e. either acid, base, salt, solvent, additive or disabled, depending upon the titration type being performed. The colour coding to the left of the list corresponds to the colour LED indicators alongside the titrant containers in the dispersion area. **Titrant name** - This is a title for the titrant container. Either type the titrant name or select from the scroll down list. The name entered here will be printed on the titrant report. **Concentration** - Specifies the concentration of the titrant in the titrant container. For pH titrations the concentration units will always be for Molarity (M), whilst for other titration types different units can be chosen. The concentration chosen will affect the precision of titration measurement **Note** - When titrating over certain pH ranges, the program may prompt for a second, more dilute, titrant to be used.

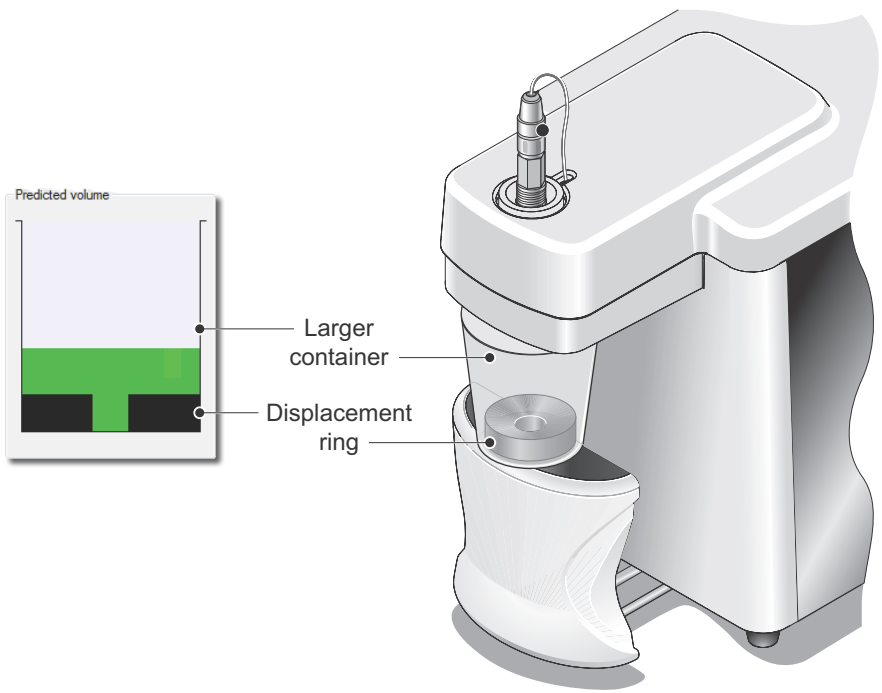
Settings	Description
Dispenser	**Automatic/Manual** - Select **Automatic** to let the software control the pump and stirrer speeds, or specify an appropriate speed for the sample using **Manual**. **Pump speed** - Sets the speed of the pump that recirculates the sample through the flow cell. For normal operation this will generally be between 30% and 80%. **Stirrer speed** - Sets the stirrer speed. Care should be taken not to introduce bubbles into the measurements by setting the stirrer speed too high. **Recirculate between repeat measurements** - If more than one measurement is being made at one titration point then this check box gives the option of either making all measurements on the sample within the cell or recirculating the sample between measurements. This option is selectable for Size measurements, but is always on when performing pH vs Zeta potential measurements. This is to prevent the pH reading from being affected when the Zeta voltage field is applied.
Sample volume	This is used to inform the software how much sample is initially added to the sample container. The more accurately this volume is specified, the more precise the titration will be. The initial start volume will vary according to the titration type being performed; generally this will be between 3ml minimum and 25ml maximum. When the magnetic stirrer is used a minimum of 8ml is necessary for pH titrations, and 5ml for all others.
Filter	This option should be enabled when running the Titrator with a filter fitted. This is to ensure correct operation of the pump. With the **Filter is fitted** checkbox enabled, the pump reverse action is disabled to avoid dislodging particles from the filter. Extra pressure from reverse pumping when the filter is attached may create leaks within the system.
Predicted volume	On the left of the **Titrants** dialogue a display is shown indicating how full the sample container will get. This display estimates the predicted total volume that will eventually end up in the sample container (initial sample volume plus all the titrant additions), to ensure that the container does not overflow and is full enough to cover the dispense tubes. The display indicates by colour the volume and risk level of any overflow. Unless a dilution titration is being performed (see below) the display will always show the 'standard' sample container.

Settings	Description
Predicted volume (continued)	The **maximum** volume of the 'standard' sample containers supplied by Malvern is 25ml. ■ **No risk**: The display will show **green** if the predicted volume is within the range **5** to **15ml**. ■ **Low risk**: The display will show **amber** if the predicted volume is **greater** than **15ml**. ■ **High risk**: The display will show **red** if the predicted volume is **greater** than **20ml**.

Note
The predicted volume is only an estimate. It is least accurate for pH titrations as buffering is difficult to estimate.

Performing dilution titrations:

For dilution titration ratios greater than 4:1, the **Predicted volume** container graphic in the dialogue will change to show that the large-volume containers should be used. This is due to the larger amounts of titrant that will be required to perform the dilution.



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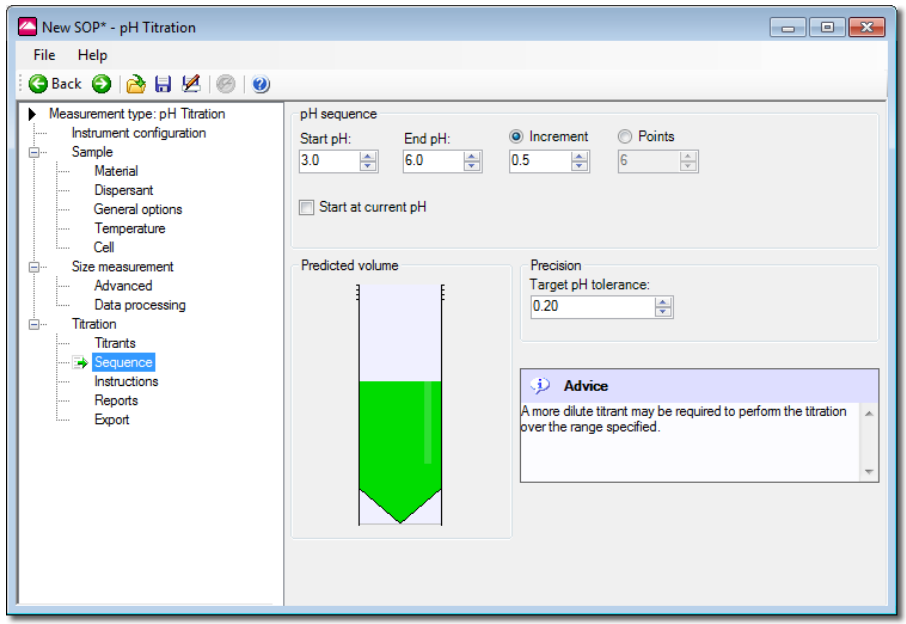
When using the large-volume containers the **displacement ring** should be placed at the bottom. The displacement ring will displace the small initial sample upwards, within the centre of the container, and towards the titrant and sample tubes. This minimises the amount of sample necessary to start the dilution, and ensures the magnetic stirrer is positioned correctly. The sloping top of the ring guides the tubes into the centre of the container, whilst also preventing any sample from resting on the top of the ring.

The displacement ring will also as act as a weight to keep the container in place when resting on the magnetic stirrer.

A message will appear to indicate that **both** the sample and titrant containers used must be the large volume type.

Titration - Sequence

This dialogue enables the titration measurement details to be selected, such as the number of points, the range of the titration and the equilibration time. Depending upon the titration type selected - pH, dilution or additive - the dialogue will alter accordingly. The same format will be used, with generally just the text and titration units altered to reflect the titration chosen. The dialogue shown is for a pH titration.



Settings	Description
pH/ Concentration sequence	The sequence area enables the **Start**/**Initial** and **End** pH or concentration values to be set. The display will change depending on the type of titration set in the measurement type dialogue. **Start/End pH** - With a titration selected, enter the **Start** and the **End** values in the respective boxes. At the start of the titration the titrator will change the pH or concentration of the sample to match that requested for the start point. **Initial concentration** - For additive and dilution titrations only. During a pH titration the pH probe measures the current pH value of the sample; but, as there is no facility to read the current concentration of the sample on the MPT-2, a value must be specified here stating the concentration of the sample before any titrants are added. With the dilution titration the concentration specified will be that of the sample container. The **End concentration** will be the desired concentration level required after dilution has finished, the dilution being performed in the number of points (steps) specified. **Increment**/**Points** - Change the **Increment** value to determine how many titration points will be measured. The number of points to be measured will be shown alongside. Increment (**points**) is used for defining the number of different sample conditions. A maximum of 30 points can be measured in a single titration. Increment is not available for Additive log or Dilution titrations. **Start at current pH / initial sample concentration** - Selecting this check box will override the **Start** value. The titrator will then use the current value and equally space the titration points between this value and the **end setting**. The **Start** value box now will be greyed out but will indicate the current pH or concentration value.
Precision	This area is **only** displayed for pH titrations. The sample may have a buffering effect that will cause the pH to drift during measurement. The sample must therefore be allowed to equilibrate to a stable value before measurement. This is achieved by selecting a **Precision** tolerance value. This value will allow a balance to be made between performing a slow titration and obtaining points close to the requested value (low tolerance values), or a faster titration where the points are slightly different to those selected (higher tolerance values).

Settings	Description
Predicted volume	Refer to description under the **Titrants** SOP dialogue.

Titration - Instructions

See the description in the Zetasizer Nano manual.

Additionally a prompt is available to instruct the operator to perform a clean sequence before the measurement begins.

Starting a Titration SOP measurement

The system is now ready to start the actual measurement.

To start an SOP measurement, select **Measure-Start SOP**. The **Open SOP** dialogue will appear. Select the SOP that will be used and select **Open**. If an SOP has not been specified for the sample, create one as described in the previous section.

The titration measurement will then follow the measurement sequence as detailed in the Zetasizer Nano manual.

- Any necessary pre-measurement instructions will appear.
- This may be followed by a **Labels** dialogue, allowing the measurement to be named and any other information about the measurement to be entered. Select the **OK** button when ready.
- The **Measurement display,** discussed below, will now appear.
- Follow the instructions on the status line of the measurement display and press the **Start** button to start the measurement.
- The measurement sequence will complete and the results can be viewed - see **Displaying the results.**

Measurement sequence

Note
The status bar will prompt for certain actions during the course of the measurement.

A titration measurement performs exactly the same processes that are done when performing a standard measurement, plus the inclusion of titration specific steps.

For details of the standard measurement sequence refer to the Zetasizer Nano manual for details.

The actual measurement sequence will depend upon the measurement being performed, though in general an autotitration measurement sequence will be as follows:

1. With the sample container and the cell inserted, **Start** is pressed. The cell is first checked to identify the cell type fitted, and that it agrees with that selected in the SOP. Once identified the measurement sequence continues automatically. The pump and stirrer will be set to the values requested in the SOP and the pH value will be read.
2. The titration will now start at the value specified with in the SOP. This will either be the "**Start**" value or "**..at current value**".
3. If **Start at current value** was selected the system will immediately go into performing a measurement. If this was not selected the unit will add titrants to the sample to bring it to the start value specified.
4. Once the start value has been obtained a measurement will be performed. The sample will recirculate if specified.
5. With the first measurement complete more titrants will be added to the sample to bring it to the second measurement point, where another measurement will be performed.
6. The measurement sequence continues until all points have been measured.
7. The measurement sequence will now complete and add a result to the current open file.

The progress meter indicates the measurement progress during each stage.

Note

If **Close** is pressed while a measurement is in progress the screen will close, but only the data for the currently performed titration measurement will be lost. The data from all completed titration measurements will be preserved; i.e. if 15 individual measurements are to be performed for one complete titration, and the titration is closed on measurement 10; the first nine measurements data will be preserved, but the 10th measurement data will be lost.

For Zeta potential measurements, when the titration measurement is closed before all individual measurements are completed, no isolelectric point will be generated.

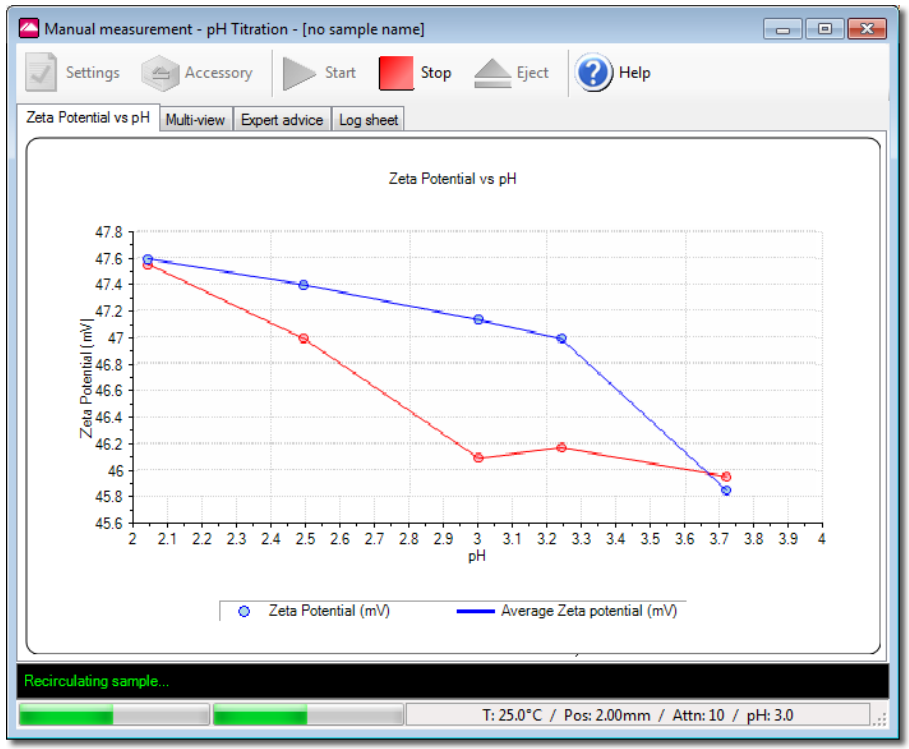
The 'Titration' measurement display

The Titration measurement displays are virtually identical to those shown when performing a standard Zeta or Size measurement. The only difference being the inclusion of a “measurement type versus titration” tab, i.e. **Zeta/Intensity vs pH** as shown below.

The standard tabs are explained in the Zetasizer Nano manual.

Titration/measurement type (pH/Zeta example)

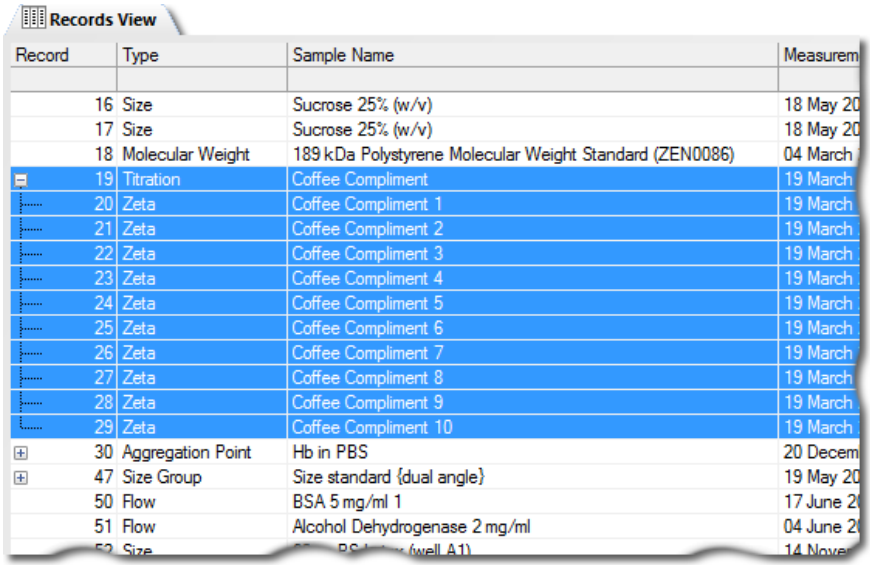
This tab details the titration measurement against the measurement type that is simultaneously produced. The graph shown will only appear after the measurement sequence has finished, and show a plot of titration v measurement type, i.e. pH v Zeta potential.



The graph above shows a plot of the titration performed, with each titration measurement point indicated on the graph. A **weighted average** line joins the **mean** measurement result obtained from each group of measurement points.

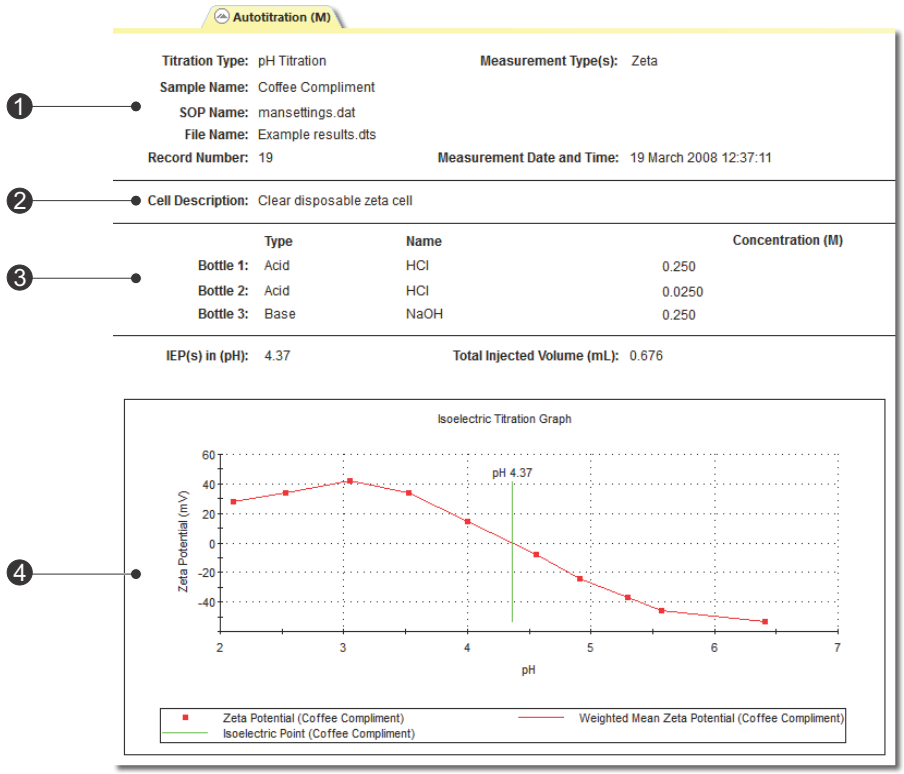
Displaying the 'Titration' measurement results

To display a titration report, select a 'Titration' type measurement record and then select the appropriate **report tab**. The report will show all appropriate measurement information for that record.



When a Titration result is selected in the Records view, all individual associated measurements will be selected too. These correspond to the number of titration points measured during the complete titration. For example, if a Zeta titration measurement used 16 titration points, then 16 Zeta measurements records will be shown directly underneath the prime titration record.

The standard report for titration measurements is shown below. The particular graph displayed is a **pH v Zeta potential** plot.



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The report is divided into four areas; these are described below. For different titration measurements the details shown will change.

① Sample

The Sample section gives details of the sample parameters. This includes the sample name, record number, measurement date and time, dispersant name, the SOP used and the measurement file name. The information shown is generally that which was inputted into the SOP measurement dialogues.

② Cell

The Cell section gives details on the Cell type used during the measurement.

③ Titrants

The Titrants section displays the same information specified in the Titrants SOP dialogue:

The **Titrants** Configuration area is used to set up the features of the titrants used, such as its type, name and concentration.

- **Type**.
  Displays the type of titrant in each of the containers; either acid, base, or disabled.

- **Name**.
  This is a title for the titrant.
- **Concentration**.
  Specifies the concentration of the titrant, this is usually between 1.0M and 0.01M. The concentration chosen will affect the precision of titration measurement.

④ Results

The results are shown in graphical form.

The graph shows a plot of the titration performed, with each titration measurement point indicated on the graph. The **Total injected Volume (ml)** is indicated at the top of the graph. This is the amount of titrant injected into the sample during the course of the measurement.

For Zeta potential measurements the **Isoelectric point** is also indicated at the top of the graph. This is the pH point at which the Zeta potential is zero.

Please see the **Size** description in the Zetasizer Nano manual for details on altering the graph.

Manually controlling the Titrator

A manual measurement is where all the measurement parameters are set immediately before the measurement is performed. This is ideal if measuring many different types of sample, or experimenting with the measurement parameters and different levels of titrant addition.

To perform a manual measurement, select **Measure-Manual** from the menu bar. A manual measurement dialogue will appear where the measurement settings can be chosen and, if required, saved as an SOP. Once chosen, the measurement can begin by simply pressing the **Start**  button on the **Measurement display**.

Manual control

There are two ways to control the MPT-2 Titrator accessory manually, these are:

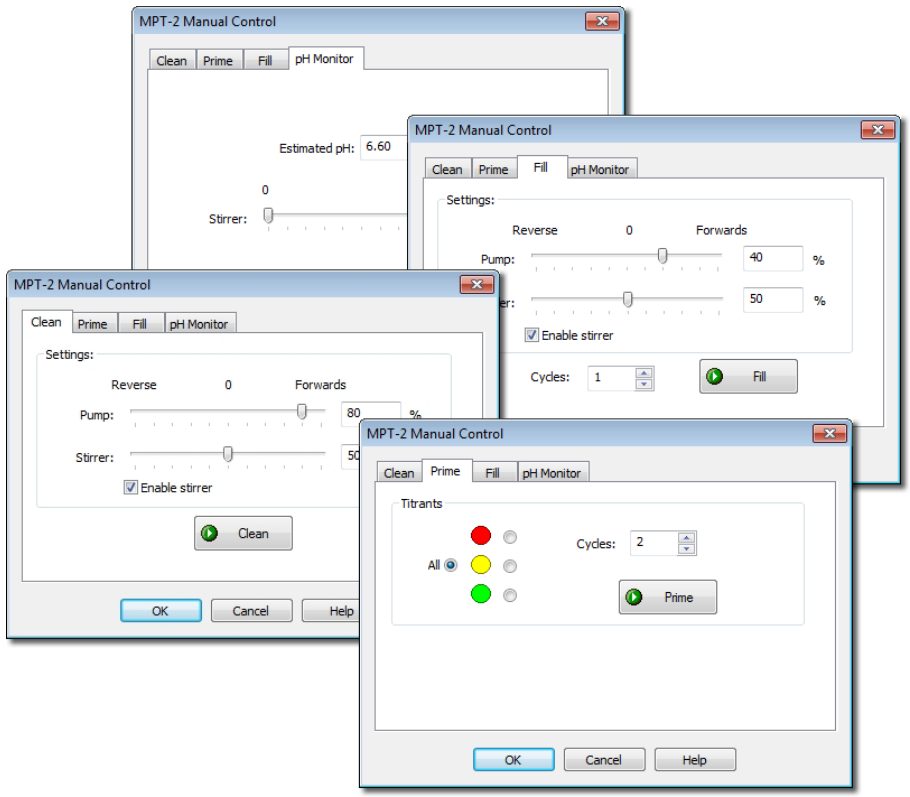
- The **Manual accessory control** dialogue.
- The **pH calibration** dialogue.

The Manual control dialogue

The **Manual control** dialogue is used to clean and prepare the unit prior to a measurement. This includes setup instructions such as priming, cleaning and calibrating the pH probe.

The **Manual control** dialogue is displayed by selecting **Tools-Instrument-MPT-2 Titrator- Manual control** within the Zetasizer Nano software, or by selecting the **Manual control**  icon.

The different tabs, **Clean**, **Fill**, **Prime** and **pH monitor**, and their functions are described below.

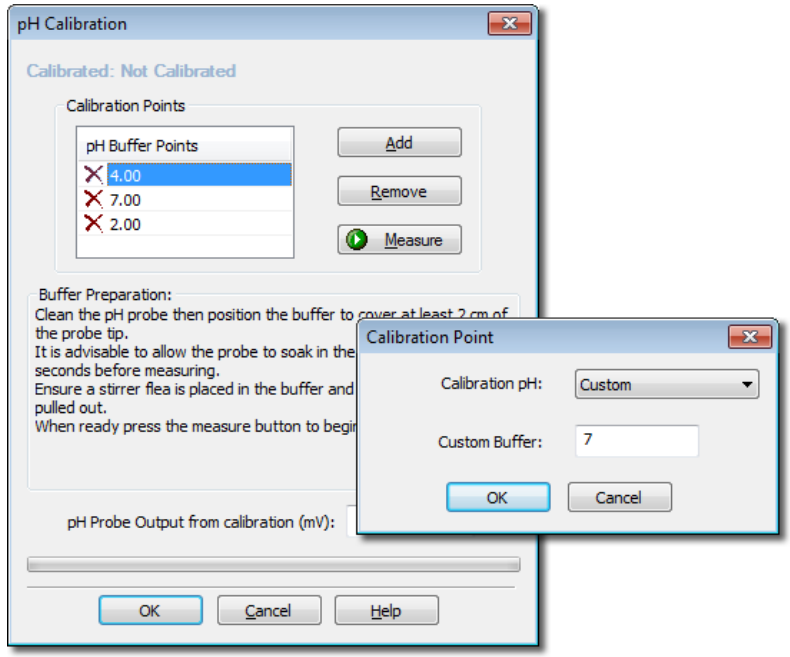


It should be noted that all prime, fill and clean quantities are determined by the number of cycles performed, each cycle performs either one complete fill, prime or clean operation.

Option	Description
Clean tab	The **Clean** tab enables the sample tubing and cell to be flushed and cleaned. **Settings (Pump and Stirrer**). These allow setting of the stirrer and of the pump to circulate the cleaning fluid through the flow cell. The slider bar or value box can be used to select the speed and direction; each control will alter to reflect the other's setting. **Enable stirrer** will turn the stirrer on or off. Refer to the **Maintenance** chapter for instructions on use.
Fill tab	The **Fill** tab is used to fill the sample tubing and cell prior to beginning a measurement. This primes the cell in the same way as the titrant tubes below. The pump and stirrer controls work in the same way as described in the Clean tab section. Refer to the **Connection and preparation** chapter for instructions on use.
Prime tab	Priming is the process of ensuring that all the titrant delivery tubes are free of air and full of liquid. Generally this is done after changing titrants or after the system has not been used for a time, e.g. overnight. Each titrant delivery tube can be individually primed, or **all** tubes can be primed simultaneously. The coloured indicators on the dialogue match the LED indicators next to the titrant container, and also the titrant tubing connectors. Refer to the **Connection and preparation** chapter for instructions on use.
pH monitor tab	This tab continually reads and displays the current pH of a sample; either in the sample container fitted to the titrator or a separate container that the probe is inserted into. The reading will update every second. If using a separate container, place the probe in the sample, then wait for the reading to stabilise before reading the value. **Note**: It is good practice to stir a sample during a pH measurement.

pH calibration dialogue

With this dialogue the pH calibration points can be set and the calibration performed.



It is important that the pH probe is correctly calibrated on a regular basis. If the probe is not calibrated then the points of the measurement may be incorrect.

It is recommended that the calibration should be performed at the beginning of each titration session.

The **pH calibration** dialogue is displayed by selecting **Tools-Instrument-MPT-2 Titrator- pH calibration** within the Zetasizer Nano software, or by selecting the **pH calibration** icon. Brief instructions on performing the calibration appear on the dialogue.

Calibration points can be added or removed using the respective buttons. Adding a pH point will display the **Calibration point** dialogue; input a pH point and press **Ok** to add it to the **calibration points** list.

Calibration can proceed by simply selecting the pH buffer points to use and pressing the **Measure** button. Guidance instructions on performing the calibration will appear on the dialogue.

A **progress bar** shows how far the calibration routine has proceeded. A tick is displayed once a pH point has calibrated.

Also displayed in the dialogue is the **pH probe output from calibration (mV)** value. This is a direct voltage reading generated by the pH calibration circuitry and corresponds directly to the **pH point from calibration** value alongside. During the calibration routine this value should constantly change. If it does not then the probe is either not immersed sufficiently in the pH solution, or the probe is broken or disconnected.

While a pH calibration is being performed **pH probe output from calibration (mV)** will change to **pH probe output (mV)**, and **pH point from calibration** will change to **Estimated pH**.

To calibrate the pH probe the follow the instructions in the **Maintenance** chapter.

Vacuum Degasser - Operational guide

Degasser specific Health and safety

Warning!
Use appropriate care when handling flammable solvents. Make sure that there are no leaks in the titrant lines - refer to **General Operation - Initial pump-down**. Ensure that hazardous exhaust gases are properly vented.

General Operation

Powering up the Vacuum degasser

With the Vacuum degasser plumbed into the system and the power lead installed, as described in the, turn on the rear panel power switch. The green Power LED should illuminate.

Immediately upon turning on the instrument, the microprocessor examines the vacuum sensor signal to confirm that it is within an expected range. Following the start-up test, the microprocessor ramps the vacuum pump to high RPM, to quickly exhaust atmosphere from the vacuum chamber. As the vacuum level approaches the preset control value, the pump RPM will slowly ramp down to a low speed (typically 40 to 60 RPM) and will vary slightly as needed under the changing degassing load to maintain a virtually constant vacuum level.

Initial pump-down

During initial pump-down, the yellow Status LED will be lit. Once the vacuum has reached normal operating level, the yellow LED will extinguish and the green vacuum LED will illuminate. As well as checking the front panel LEDs, the running of the vacuum pump can be checked by placing a hand onto the instrument and checking for vibration

Start the titrant flow through the system and check for leaks around the connectors.

Note
If a leak occurs at the connection, tighten the fitting an additional 1/8 turn. If the leak persists, disconnect the leaking fitting and inspect it. If the nut and ferrule appear to be in good condition, reconnect the fitting. If the leak persists, replace the nut and ferrule and repeat the procedure until you achieve leak-free operation.

The Vacuum degasser maintains a constant vacuum pressure of 50 mm Hg absolute (nominal) by varying the speed of the vacuum pump as needed depending on the degassing load in the system. The pump is designed for at least 5 years of constant running and has integral in-pump venting, which eliminates the need for stop-start running (U.S. Patent 6,248,157). The vacuum level and pump speed are constantly monitored by the microprocessor for changes in operating conditions which might be attributed to chamber internal leaks. If a potential leak is detected (if the pump RPM > 300 for 30 minutes), the pump will be shut down and the yellow Status LED will flash. The vacuum is maintained as long as the degasser is powered on. Titrants flowing through the degasser will continue to be degassed so long as the instrument is on and running.

Also refer to **Smart leak detection**, later in this chapter.

Turning off / powering down

Turn off the Vacuum degasser when the titrator to which it is connected is not in use. The vacuum chamber(s) will slowly return to atmospheric pressure when the unit is powered off. This is accomplished by a small, in-line vacuum bleed and reduces the possibility of titrant vapours condensing in the vacuum tubing or pump head.

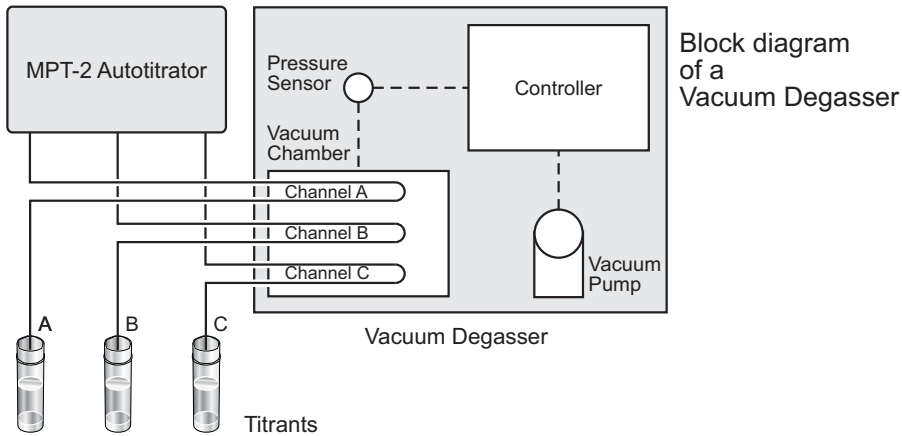
Flushing - cleaning the degasser

When flushing a line of titrant, the tubing inside the degasser contains a very small amount of titrant (approximately 480 microlitres). When changing from one titrant to another; if the final titrant is immiscible with the first, use an intermediate titrant that is miscible with both the initial and final titrant. Once air bubbles have been cleared from the titrant line, any further bubbles observed will be coming from the titrant container or from a leaking fitting.

Note
The MPT-2 Titrator should be primed after the degasser has reached equilibration. This will ensure that the titrant in the tubes from the degasser to the sample tube is fully degassed. The Vacuum degasser uses Teflon AF® membranes; which fully degas titrants in the time it takes for the volume to pass through the chamber.

Principles of operation

The Vacuum degasser consists of a vacuum chamber, degassing tube, variable speed vacuum pump, microprocessor controller, sensor, and check valves. The titrant flows into a degassing tube, which is inside a vacuum chamber. Decreased pressure in the chamber causes the outward movement of gas, dissolved in the titrant, across the tube wall, thus degassing the titrant; this is in accordance to Henry's Law. The pressure in the vacuum chamber is established by the vacuum pump and monitored by the microprocessor through an integrated absolute pressure sensor. Degassed titrant exits the vacuum degasser and enters the pump.



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Smart leak detection

An additional benefit of maintaining a constant vacuum level is that a potential leak in the vacuum degassing system can be observed by monitoring the RPM of the pump. If a leak occurs within the chamber, the microprocessor will increase the pump RPM in an attempt to maintain the vacuum level. If the pump cannot maintain the vacuum level (if it runs at an elevated RPM for more than 30 minutes), the yellow LED will flash, indicating a possible leak condition, and the system will shut down and go into a "safe" mode.

Principles of degassing using Teflon AF® membranes

This relatively recent addition Teflon AF® membranes to the field of degassing is due to their having properties not found in other fluoropolymers. The fully amorphous nature of this fluoropolymer and its molecular structure creates a molecular level porosity unlike the mechanically induced porosity in PTFE extruded tubing. In addition, unlike the process used in extruding PTFE, no extrusion agents are

needed (like kerosene, etc.) which contaminate titrants until they are extracted by the titrant over time. Likewise, this molecular structure, combined with the very small surface areas required to degas the titrant, reduces the possibility of carryover from one titrant to another to virtually zero.

Teflon AF® is so non-polar that it is both solvophobic and hydrophobic. This feature of Teflon AF® reduces the possibility of cross-channel contamination from one channel to another, and when combined with the ultra-low internal volumes of Teflon AF® channels needed for low flow rates, all but eliminates any cross contamination concern. Teflon AF® has been used in certain optical systems associated with HPLC for a few years without concern for normal HPLC solvents. However, Teflon AF® is soluble in certain solvents (Refer to the **Chemical compatibility** section in the appendices chapter) and **must** not be used to degas these solvents when used for titration.

Teflon AF® is permeable to some degree to water vapour whereas PTFE is not. While the vacuum pump in the Vacuum degasser contains internal provisions for sweeping water or titrant vapour from the pump continuously, it is possible that over time, high concentration buffers may form crystals within the channel due to the loss of water within the channel. The same precautions should be taken to prevent crystallization within these channels as are taken for the Titrator. Refer to the **Short-term shutdown** procedures in the **Maintenance** chapter.

Extending the degassing flow rate range

Certain organic titrants outgas upon mixing with water, if not properly degassed. These titrants are generally alcohols (e.g. methanol), acetonitrile and tetrahydrofuran. Passing water and methanol through a single channel is generally sufficient to degas these titrants so outgassing does not occur upon mixing when a 75% methanol: 25% water mixture is generated by the Vacuum degasser or pump at a flow rate of 2 mL/min. If outgassing does occur, or if a flow rate higher than 2 mL/min. is required, it is a general rule that only the organic portion of the titrant needs to be passed through a second degassing channel to ensure outgassing does not occur. This is due to the ability of all organic titrants (e.g. methanol) to hold at least 10 times more dissolved atmosphere than water can.

To more thoroughly degas a titrant, connect the outlet of the organic channel to the inlet a second channel and the outlet of the second channel to the pump. This places the two channels in series and doubles the degassing capacity for the organic portion of the titrant.

Shutdown

There are two types of shutdown procedures: **long-term** and **short-term.** These are explained in the **Maintenance** chapter.

Maintenance

MPT-2 Titrator maintenance

Caution!
Only a qualified Malvern representative, is allowed access to the inside of the titrator. There are no user serviceable parts inside the titrator.

The titrator has been designed so that supervisor/operator maintenance is kept to a minimum. This section explains the routine maintenance procedures that the supervisor/operator can perform. These procedures are:

- Cleaning the titrator.
- Maintaining the pH probe.
- Replacing the Dispensing syringes and o-ring.
- Changing the filter and tubing

A summary of how often these procedures should be carried out is detailed in the maintenance schedule later in this chapter.

Additionally, this section contains a list of parts and part numbers of consumables and items that can be replaced by the user in the event of failure.

Cleaning the accessory

Caution!
The surfaces of the system may be permanently damaged if samples or titrants are spilt onto them. If spillages should occur, then the system should be disconnected from the power supply before scrupulously cleaning any spillage.

Cleaning the Titrator surfaces

Periodically, the covers should be thoroughly cleaned using a mild soap solution on a damp cloth. Always ensure that the accessory is disconnected from the power supply before cleaning.

Never use excess liquid to clean the accessory and always avoid the electrical components on the rear panel.

Always ensure that the accessory is completely dry before applying power.

Never use a solvent-based solution to clean the accessory as damage to the painted surface may result.

Cleaning the sample and titrant path

To avoid cross-contamination of samples, the sample flow path should always be kept scrupulously clean. Always flush the sample after each measurement session.

Note
Choose an appropriate cleaning fluid for the sample or titrants being used (e.g. deionised water). Ensure that it will reliably clean the sample or titrants used from the tubing.

Cleaning the sample tubing:

1. Remove the sample container and replace with one full of cleaning fluid.
2. Press the **Clean** button to begin the operation.
3. The pump will circulate the cleaning fluid through the sample tubing until the **Clean** button is pressed a second time. Use the pump and stirrer controls (below) to adjust the pump and stirrer speeds during the cleaning.
4. Perform the clean a few times to ensure the sample tubing is properly cleaned, i.e. stop the clean operation, refresh the cleaning fluid and repeat. It is recommended the clean procedure is performed three times to ensure adequate cleanliness has been achieved.
5. Once started, pressing **Clean** a second time will stop the operation.

Note
The larger the cleaning fluid container the more effective the cleaning operation will be.

Note
Pumping air into the tubing prior to the cleaning fluid will aid the cleaning. This will act as a buffer, first driving the sample through the tubing before the cleaning fluid cleans the tubes. This is simply done by removing the cleaning fluid while leaving the pump running.

Cleaning the Titrant tubes

The titrant tubes can also be cleaned using the same tab. Simply insert containers of cleaning fluid in place of the titrant containers. - refer to the connection prep chapter

Maintaining the pH probe

The pH probe is essential to ensure good performance of the Titrator, and its maintenance is of paramount importance. As with any pH probe, its performance will degrade over time and will eventually have to be replaced. The frequency of replacement will be determined by; the maintenance of the probe, how frequently it is used and the types of sample being measured. In this respect a pH probe can be thought of as a "Consumable" item. Malvern recommends that the probe is replaced every year, but this frequency may vary.

Note three important points when using a pH probe:

6. The performance of the pH probe will be severely affected if it is allowed to dry out (even for short periods).
7. The pH probe should be calibrated at regular intervals.
8. The probe should be cleaned regularly - If the probe responds slowly a message to clean the electrode will be given.

This electrode operates over a range of pH 1 to pH 14, and temperature range of 0 to 70°C, though it is recommended that the titrations are performed at ambient temperature conditions, i.e. by setting the measurement temperature to equal the ambient temperature.

This is for two reasons:

1. The pH Probe is not temperature compensated, so the sample in the container must be the same temperature as the cell.
2. At higher temperatures the operating life of the pH probe will be greatly reduced.

Preparation before first use

1. Remove the screw end-cap and connect to the cable supplied.
2. Remove the membrane protection teat. This teat contains a small amount of saturated KCl solution (3.8M).
3. To allow air pressure equalization in the reference junction part of the probe, remove the rubber cover, or sleeve that covers the reference junction vent.
4. Check that the reference electrolyte is about 1cm below the level of the opening. Top up with the electrolyte supplied if necessary.
5. Check that there are no air bubbles within the inner glass membrane. Bubbles may be removed by shaking the electrode using a similar action used with a clinical thermometer.

Keeping the pH probe wet

If the pH probe is allowed to dry out, even for short periods, the performance of the probe can be severely affected. It may not be possible to recover a degraded pH probe.

Typically, when the probe is immersed in a buffer solution, the pH should be attained (to within 0.1 of the pH) within 10 seconds. If this time exceeds 30 seconds, and the probe cannot be recovered, then the probe should be replaced.

When changing the sample container, do not allow the probe to be exposed to air for more than 20 seconds. Make sure that the next sample container is ready to attach, or alternatively have a spare container of storage solution available.

pH probe storage

The pH probe can be stored in water or a soaking solution. A soaking solution is a liquid that the probe can be immersed in to prevent it from drying out. The following guidelines should be followed when using a pH probe:

- The probe can be immersed in the **sample** solution for short periods (a few hours).
- The probe can be immersed in **tap water** for the up to a day. (Do not use deionised water as it will destroy the charge equilibrium of the glass electrode).
- For long periods (more than a day) the probe should be immersed in either a dilute acid solution (pH 2 to pH 4) or an acidic buffer such as pH 4.
- If the electrode is not being used for a period of, say, more than a week, then it is advisable to protect the glass membrane by replacing the protective rubber teat containing a small quantity of the electrolyte solution. It is best to squeeze the end of the teat first to make it easier to slide on and also to prevent electrolyte being forced back into the probe.

If the probe has become dry, then it may be possible to recover the performance by soaking the probe overnight in a solution of 0.1M hydrochloric acid. However, the performance may never fully recover and there may be no alternative except to replace the pH probe.

Cleaning the pH probe

The probe should be periodically cleaned. The calibration procedure detailed below will indicate whether the probe requires cleaning.

Chemical cleaning is usually sufficient. To chemically clean, soak the probe overnight in a solution of 0.1M hydrochloric acid, then use a soft tissue soaked in isopropanol and wipe over the end of the probe. Once cleaned, re-calibrate the probe to see if its performance has recovered.

Some probes have a PTFE 'wick' which is fibrous. This is more easily wetted than the ceramic junction but can become clogged by very fine particles in the dispersion.

When this happens, the pH probe will not respond to pH changes in the sample. Carefully brushing this wick can recover performance.

This effect is reduced by using more dilute samples.

Calibrating the pH probe

The performance of the titrator relies on the pH probe being correctly calibrated. If the pH probe has not been calibrated then the pH values will be incorrect.

It is recommended that the pH probe is calibrated before each titration session.

To calibrate the pH probe the following is needed:

- At least two pH buffers. The default buffers are pH 4 and pH 9 (these are supplied with the probe). See **Choosing the buffers when calibrating** later in this section for advice on selecting buffers. A maximum of four buffers can be used for the calibration.
- A container of clean deionised water to rinse the probe between measurements.

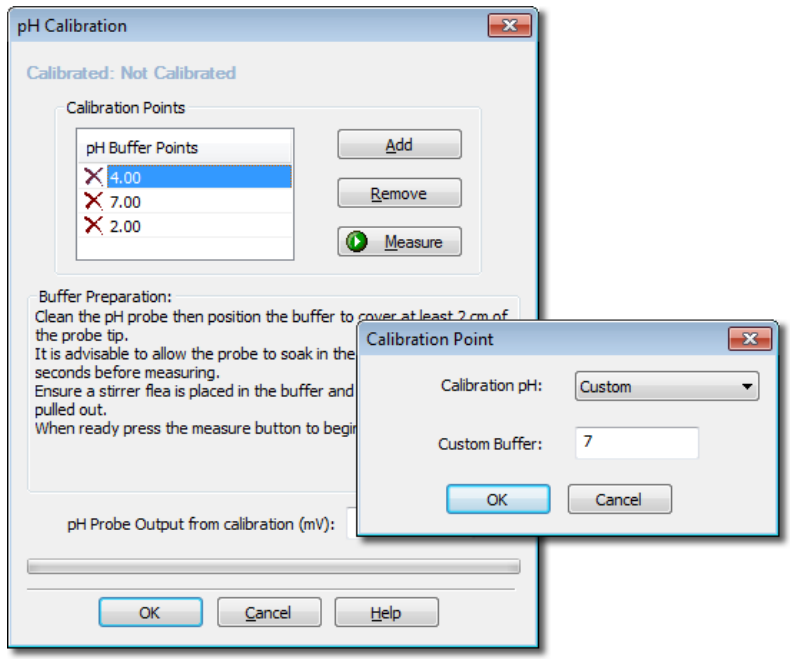
Ideally, the pH probe should be calibrated while fitted to the system and using the standard sample containers.

Note
The pH probe should be recalibrated if the room temperature changes by more than 5°C.

Calibration

Calibration is performed by selecting a calibration point that will match the pH buffer to be used, and pressing the Measure button. Instructions are present on the calibration dialogue to give guidance through the process.

1. Rinse the electrode in deionised water and dab dry with a good quality lens tissue (one that does not shed fibres/lint free).
2. Open the **pH calibration** dialogue by selecting **Tools-Instrument- MPT-2 Titrator- pH calibration** within the **Zetasizer Nano software**, or by selecting the **pH calibration** icon.



3. Select the first calibration point: press the **Add** button and enter the calibration pH required. Press **OK**. This point will now be added to the calibration point list.
4. Repeat the above for all the required calibration points, i.e. pH 4, 7 and 9. The chosen pH points will show an alongside. Default pH points will be pH 4 and pH 9 as these are the buffers originally supplied with the pH probe. See **Choosing the buffers when calibrating** later in this section for advice on adding and selecting buffers.
5. Select the first pH point and place the pH probe into the matching pH buffer, wait for the **pH probe output (mV)** value to stabilise and press the **Measure** button. The software will then check that the pH reading is stable before con-

tinuing. (After a pH calibration has finished, **pH probe output (mV)** will change to **pH probe output from calibration (mV)**.

6. When the calibration of the first pH point is complete, the pH point will show a ✓ alongside.
7. Remove the probe from the first pH buffer, rinse with deionised water and dab dry.
8. Continue the calibration until all required pH points have been calibrated.

During the calibration a **progress bar** at the bottom of the dialogue indicates how far the calibration routine has proceeded.

The ticks or crosses alongside the pH probe indicate the following:

✗	A pH point has been chosen but not yet calibrated.
✓	A pH point has been chosen and been calibrated.
✓?	Calibration points selected during a previous calibration, which have not been calibrated during the current session. If required select these points and perform the calibration process as described. A tick will appear once it is complete.

Points can be deleted by selecting the point and pressing **Remove**.

Choosing the buffers when calibrating

The default buffers for calibrating are pH 4 and pH 9. Other values may be used but ensure that the lowest and highest values are at least 3 pH values apart. Also if all titration measurements tend to be in the lower end of the pH table it is advisable to use pH 2 and pH 5 buffers to calibrate the probe.

Adding a pH buffer point

To use a different pH value, a new pH buffer point must be added to the selection list. Press **Add** to display the **Calibration point** dialogue, then perform either of the below actions:

1. Select a defined pH point from the **Calibration pH** list and press **OK** to add it to the **Calibration points** list in the main dialogue.
2. Or if using a pH buffer not available in the list, select **Custom**, type the value in the **Custom buffer** entry box, then press **OK** to add it to the **Calibration points** list in the main dialogue.

If the maximum accuracy of calibration is required over a wide pH range, then 3 or 4 buffers should be used

The pH probe for the Titrator is supplied with sachets of pH4 and pH9 buffers. Follow the instructions supplied with the sachets for the best methods of making the buffer solutions.

As buffers are typically low cost items, it will be more economical to source from a local supplier. If finding a convenient source proves difficult, contact the local Malvern representative who will be able to give advice.

Common Problems

pH electrodes are versatile but their performance is dependent on the sample being tested. For example, pH measurement of high purity water or liquids containing sulphur ions will require special electrodes.

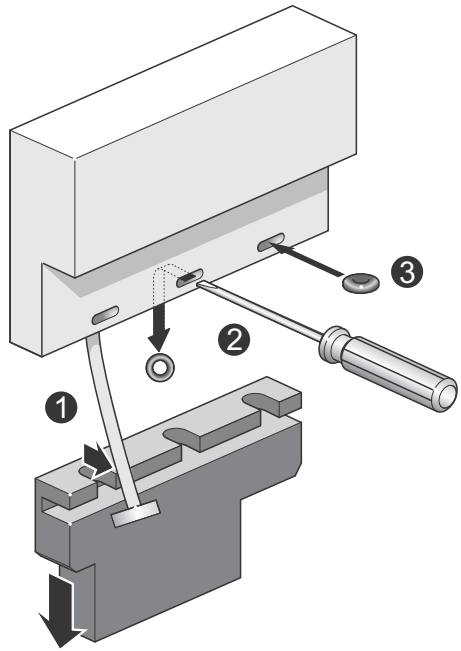
- Slow response can often be caused by the build-up of deposits on the glass membrane or frit. Initially, try cleaning the probe by dipping into concentrated Nitric Acid for 10 seconds. If this is unsuccessful, repeat for one minute. If still unsuccessful then regeneration may be possible by dipping the membrane for one minute into a solution of 2% HF and 5% HCl whilst stirring. Should the frit be blocked by proteins then try soaking the electrode in an appropriate organic solvent, or a solution of 10% pepsin in 1M HCl. As such actions usually dehydrate the glass membrane, it will be necessary to soak overnight in 3M KCl solution. If these actions are unsuccessful then the electrode will need to be replaced.
- Always ensure that the level of the electrode's electrolyte is above that of the liquid under test. This will prevent electrolyte contamination, and ensure a good electrical contact. With the blue rubber cover removed, the electrode uses a small amount of electrolyte per day. When in continuous use, weekly top-ups are recommended.

Replacing the dispensing syringes and o-ring

It is recommended that all three dispensing syringes and o-rings are replaced at least every 12 months or sooner if leaks develop. Replacement syringes and o-rings are available through the Malvern representative (see **User consumable and spares** at the end of this section).

To remove a syringe and o-ring:

1. Turn off the accessory and remove the side cover.
2. Manually push down the syringe mount.
3. Pull the base of the syringe ① out of the syringe mount, and then out from the o-ring.
4. Using a small screwdriver push the o-ring out of the manifold assembly ②. The o-ring will fall out the back of the manifold onto the base of the dispensing area.



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Replacement is the reverse of the above procedure.

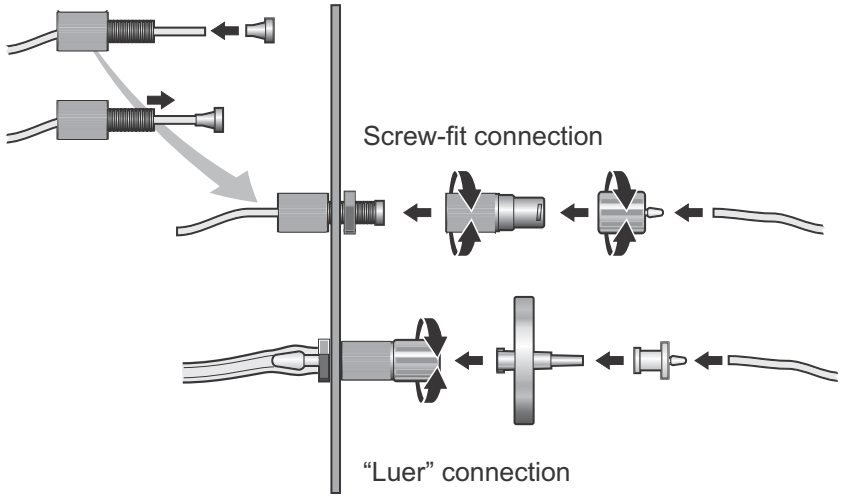
1. Push the o-ring into the recess on the manifold ③.
2. Slide the syringe mount up, checking that the syringes smoothly enter the manifold assembly - ensure the syringe does not bend severely.
3. Prime the system once all the syringes have been replaced.

Changing the filter and tubing

The filter removes all particles above the grading size of the filter fitted. To maintain optimum filtration it is recommended it should be replaced regularly. Replacement of the filter and any tubing on the titrator is performed using very similar techniques.

Generally, all internal tubing is connected with the screw-fit connections and all external tubing and filters use a 'Luer' style. The diagram below shows how the connections are made. Some connections differ slightly but the principle is the same.

To replace the tubing on the Sizing flowcell, simply pull the used silicone tubing off the harder PTFE tube and replace with new tubing. Connect the tubing at the tubing connection bracket as shown below.



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Maintenance schedule

The following maintenance schedule should be followed to ensure the accessory continues to correctly function.

Procedure	Period
Priming the system.	Every morning or at the beginning of a new measurement session.
pH probe calibration.	Preferably before each titration session.
pH probe cleaning.	Once a week or according to in-house QC procedures.
Cleaning the covers.	Once a month.
Dispensing syringe and o-ring replacement.	The three dispensing syringes should be replaced every 12 months.
pH probe replacement.	The pH probe should be replaced at least every 12 months.

Vacuum degasser maintenance

To experience long and trouble-free performance with the Vacuum degasser, it is recommended that both routine and preventive maintenance procedures be regularly performed.

Shutdown

There are two types of shutdown procedures: long-term and short-term.

Short-term shutdown (Overnight and weekends)

Observe all precautions pertaining to hazardous titrants and/or those titrants that form harmful deposits or by-products.

- Remove harmful titrants from the degasser and other instruments in the Zetasizer system.

Caution!
Damage caused by precipitating buffer salts in capillary tubing, or damage resulting from this condition, is specifically excluded from warranty.

- Flush buffer salts from the system with water by priming the titrator with demineralised water in the titrant container. Evaporation leaves salt crystals that may form harmful deposits. Remove chloroform or titrants that can decompose to form hydrochloric acid from the system.
- For weekend storage flush 60/40% MeOH/Water through the degasser, then turn off the degasser.

Long-term shutdown and storage

- Follow the first two Short-term Shutdown procedural steps above.
- Remove the column and direct the pump output tubing to a beaker. Flush the degasser, first with water and then with Isopropanol.
- Turn off the degasser. Then disconnect the tubing between the degasser and titrant containers, and the degasser and the titrator. Plug all of the ports on the degasser.
- Store the degasser in a clean, dry location.
- Before using the degasser, completely purge it with the correct titrant before reconnecting the titrator and restarting the system.

Preventative Maintenance

Perform preventive maintenance to ensure that your Vacuum Degasser will perform consistently at an optimal level. To maintain the Vacuum Degasser in the best condition, the following measures are recommended:

- Adhere to standard laboratory cleanliness practices.
  Use only high-purity titrants - preferably High Performance Liquid Chromatography (HPLC) Grade. Water should be bottled HPLC Grade, or filtered and deionised tap water.
  Filter all titrants to prevent particulate contamination and tubing blockages.
- Use only high-purity gases when drying contact areas.
  Follow the short- and long-term shutdown procedures that are described above.
- Routine cleaning of the external surfaces of the instrument can be done using a clean, damp cloth. Immediately clean any spills which occur on or near the instrument using methods appropriate for the type of spill. Some titrants can damage the appearance and function of the instrument.

Routine Maintenance

Routine maintenance is defined as replacing the normal wear items when degradation in performance is noticed.

Consult the Troubleshooting Guide, below, if a problem has arisen.

Warning!
Never remove the Degasser's cover. There is nothing inside that requires customer service or maintenance.

Troubleshooting Guide

Problem	Probable Cause	Solution
Power switch is on, but all 3 LEDs are off, indicating no power to the degasser.	The AC Adapter is not plugged into the AC wall socket.	Plug the AC Adapter into the AC outlet.
	Blown fuse.	Contact your Service Representative.

Problem	Probable Cause	Solution
Yellow Status LED is on steadily, pump is running and RPM seems high.	Pump is in initial pull-down phase or system's degassing demand has increased.	Typically normal operation, although if pump speed continues to rise for an extended period of time (as heard by the pitch of the stepper motor) it could indicate a potential fault condition.
Yellow Status LED is flashing approximately 1 second off, 1 second on. Vacuum pump is not running.	Possible system leak.	Contact your Service Representative.
Yellow Status LED is flashing approximately 2 seconds off, 1 second on. Vacuum pump is not running.	Possible sensor or Control Board fault.	Contact your Service Representative.
Is there a way to check whether the system is operating correctly when Power and Vacuum green LEDs are illuminated, but pump can't be heard running?	Due to the design of the pump and degasser, the pump is virtually silent at low RPM, even though vacuum is good and degassing is normal.	Place a hand on the top of the unit. A slight vibration can be felt indicating the pump is operating at low RPM.
Bubbles appear through the output tubing.	Loose fitting(s).	Tighten the input and output fittings.
No titrant flow.	If a buffer titrant was left in the degasser for some time after use, it may plug the degasser elements.	Use a different channel, or connect the channel to a beaker of the titrant without the buffer. Draw the titrant through the channel to dissolve the buffer. Do not push the titrant through the channel. If this flushing action does not work, contact your Service Representative.

User consumables and spares

This section lists all available user consumables and spares. Contact the Malvern Instruments representative, quoting the part number listed below.

Part	Part number
500 'Standard' sample and titrant containers plus caps. Maximum fill : 25ml	BEK0008
10 'Large-volume' sample and titrant containers plus lids. Maximum fill : 125ml	BEK0009
pH probe Inc: pH4 and 9 buffer sachets)	SEN0106
Dispensing syringe pack Inc: 3 syringes and 3 o-rings)	ZEN4030
Magnetic Stirrer bar (Flea).	ZEN4034
Peristaltic pump head. Inc: Pump head and tubing	ZEN4036
Connectors pack for all internal and external connections on the Autotitrator except pump (mostly1/16” fittings). Inc: 4 x manifold (red) 2 x push on barb connections 4 x manifold (green) 4 x black tubing grips 4 x manifold (cream) 1 x Luer filter fittings 4 x plastic nuts 1 x Luer caps	ZEN4037
Internal tubing pack 1 for all internal connections on the Autotitrator. Inc: 20 x tubing cones (ferrules) and 8m tubing	ZEN4038
External connections pack (to Zetasizer Nano) Inc: 15 x Male Luer lock fittings (sample in connection) 5 x Female Luer push fittings (sample out connection)	ZEN4039
External tubing (to Zetasizer Nano) Inc: 1 box (25ft) silicone tubing	ZEN4040
Displacement ring (for use with large-volume beaker)	ZEN4041
Internal tubing pack 2 Includes tubing and fittings for larger bore tubing (1/8") sizes to Dispersion head	ZEN4043
RS232 cable.	CAB0020

Appendices

Specification

MPT-2 Multi-purpose titrator

Dimensions (excluding pH probe).	Width: 170mm Height: 260mm Depth: 390mm
Weight (dispenser unit only).	5.3Kg.
Power requirements. **Power supply:**	~ 100-240V, 50-60Hz 24Vdc via external power supply
Power rating.	30VA
Recommended maximum fill capacity of standard sample container. **When filled with titrant:** **When filled with sample:**	 25ml 20ml
Modes of operation.	Automatic via Malvern software.

The accessory has been designed to be stored and operated in the following environmental conditions.

Operating temperature	+10 to 35°C (+50 to +95°F)
Storage temperature	-20 to +50°C (-104 to +122°F)
Humidity	10 to 90% (non-condensing)
Altitude	Up to 2000m
Pollution degree	2
Mains supply voltage fluctuations	±10% of nominal voltage

Vacuum degasser

General

Dimensions:	Height: 5.0 in. Width: 2.87 in. Depth: 9.81 in.
Weight	6 lb
Channels	3 independent
Degassing Process	Gas permeation through a fluoropolymer membrane
Maximum Flow Rate	10 mL/min.
Degassing Capacity	~2 ppm at 1 mL/min.
Dead Volume	~480 microliters per channel for standard channel
Materials contacting titrant/solvents	PEEK, Glass-filled PTFE, Teflon AF®, PTFE

Power

Power requirement using supplied AC Adapter	100 to 240 VAC, 1A, 50 to 60 Hz
Interchangeable adaptor plugs	4 supplied with AC Adapter Interchangeable to AC Adaptor: North America/ Japan, U.K., Continental Europe, Australia
Installation Over-Voltage Category	II (IEC 60664)

Validation Output

Signal	5 mVDC / 1 mm Hg absolute from 20 to 800 mm Hg (0.100 VDC at 20 mm Hg; 4.000 VDC at 800 mm Hg)
Accuracy	±1.0% of reading ±0.010 VDC from 20 to 800 mm Hg

Operating Conditions

Ambient Temperature	10 to 35 ºC
Ambient Relative Humidity	20 to 80 % RH (without condensation)
Altitude	0 to 2000 Meters
Indoor vs. Outdoor Use	Indoor
Pollution Degree	2 (IEC 60664

Storage Conditions

Ambient temperature	–20 to +60 ºC
Ambient Relative Humidity	20 to 80% RH (without condensation)
Altitude	0 to 12000 M

Connector and Tubing sizes

Connectors	
Material	PEEK
Size / description	1/4-28 flangeless fittings for 1/16" 'Outside Diameter' tubing
Connecting tubes from MPT-2	PTFE
Material	1.6mm x 0.8mm (Outside Diameter x Inside Diameter)
Size	

Site requirements

MPT-2 Multi-purpose titrator

Note
These requirements should be read in conjunction with the system requirements as stated in the Zetasizer Nano user manual.

Nitrogen purge specification

Warning!
A Nitrogen supply must be used in a well ventilated environment. Turn **off** the supply when not in use.

If a nitrogen supply is required then it must conform to the following specifications and flow rate conditions:

- It is important that the nitrogen supply is dry, free from oil and filtered to remove any contaminants that could affect the sample being measured.
- The flow rate should be adjustable between 2 and 20ml/min.

Vacuum degasser

Space Requirements

The Vacuum Degasser is designed to sit on a standard laboratory bench top, and is plumbed into the MPT-2 Titrator system between the titrant supply and titrator pump.

Enough space should be given to allow easy access to all components of the system.

Dimensions	Height: 5.0 in.
	Width: 2.87 in.
	Depth: 9.81 in (distance front to back)

Allow additional space both in front, to accommodate the tubing connected to the unit, and behind to accommodate the power lead.

Electrical Power Requirements

The mains power supply must be clean and filtered. If necessary, fit an un-interruptible power supply (UPS) to remove any spikes or noise. The power requirement for the Vacuum degasser is indicated below.

Power requirement 100 to 240VAC / 47 to 63Hz

Caution!
Use the correct power lead for the territory.

A set of four interchangeable adaptor plugs are is included to allow the AC adapter to be plugged into the standard electrical wall sockets in North America, Japan, the U.K., most countries in continental Europe, and Australia.

If it is necessary to replace the AC Adapter - Contact Malvern instruments about replacement.

Note
Refer to the **Health and Safety** section on the main **Zetasizer Nano User manual** for more information.

Chemical compatibility

The sample flow path in the titrator and degasser has been manufactured from materials that are considered to give the widest protection from chemical attack. However, it is important to check that any sample or titrant used is chemically compatible with the materials that they will come into contact with within the accessory.

The sections below detail all materials that come into contact with the sample and titrants in the normal operation of the accessory.

MPT-2 Multi-purpose titrator

Component	Materials
Sample/titrant containers (25ml)	Polypropylene
Sample/titrant containers (125ml)	Polypropylene
Sample container mount (i.e. dispersion head)	Polypropylene
Titrant container mount	Polypropylene
Magnetic stirrer 'flea	'Teflon'
Manifold	PEEK (Polyetheretherketone)
Manifold valves	PEEK and FFKM (seal)
Dispensing syringes	PTFE
Peristaltic pump tubing	Silicone rubber
Internal Tubing	PTFE
Tubing to optical unit and cell	Silicone rubber
Tubing connectors	Polypropylene / PVDF
pH probe	Glass
Displacement ring	PEEK (Polyetheretherketone)

Vacuum degasser

Titrants and Solvents

Use only HPLC grade solvents in all analyses.

Caution!
The degassing membrane in the Vacuum Degasser is manufactured from Teflon AF®.

Teflon AF® Solvent Compatibility is:

- Teflon AF® is inert to all solvents normally used in HPLC.
- Teflon AF® is soluble in perfluorinated solvents such as Fluorinert® FC-75 and FC-40 and Fomblin perfluoro polyether solvents from Ausimont.
- Freon® solvents will adversely affect Teflon AF®.

Use of inappropriate solvents in the Vacuum Degasser will result in the dissolution and hence destruction of the membrane.

Caution!
Use appropriate care when handling flammable solvents. Make sure that there are no leaks in the titrant lines - refer to **General Operation - Initial pump-down**. Ensure that hazardous exhaust gases are properly vented.

Corrosion

All parts that contact the titrant (solvents) are made of PEEK, Kel-F®, Tefzel® or Teflon AF®, PTFE or Glass-filled PTFE

PEEK is sensitive to Sulphuric acid and certain solvents.

Regulatory information

Disposal of Electrical & Electronic Equipment

This regulation is applicable in the European Union and other European countries with separate collection systems.

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This symbol on the product or on its packaging indicates that when the last user wishes to discard this product it must not be treated as general waste. Instead it shall be handed over to the appropriate facility for the recovery and recycling of electrical and electronic equipment.

By not discarding this product along with other household-type waste, the volume of waste sent to incinerators or landfills will be reduced and natural resources will be conserved.

For more detailed information about recycling of this product, please contact your local city office, your waste disposal service, or your Malvern representative.

CE Declaration of Conformity

The CE badge on this product signifies conformance to the following European Directives.

- EMC directive 2004/108/EC {BS EN 61326-1: 2006}
- Low Voltage Directive 2006/95/EC {BS EN 31010-1: 2010}

FCC Notice (US only)

The Federal Communications Commission (FCC) mark on this product signifies conformance to FCC regulations relating to Radio Frequency Devices. These have been satisfied by testing the product against, and being found to be compliant with:

FCC CFR 47 Part 15:March 2003.Class A digital device.

The device complies with part 15 of the FCC Rules. Operation is subject to the following two conditions:

1) This device may not cause harmful interference, and

2) this device must accept any interference received, including interference that may cause undesired operation.

Note
This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Note
Changes or modifications not expressly approved by Malvern Instruments Limited could void the user's authority to operate the equipment.

Canadian Regulatory Information

This digital apparatus does not exceed the Class A limits for radio noise emissions from digital apparatus set out in the Radio Interference Regulations of the Canadian Department of Communications.

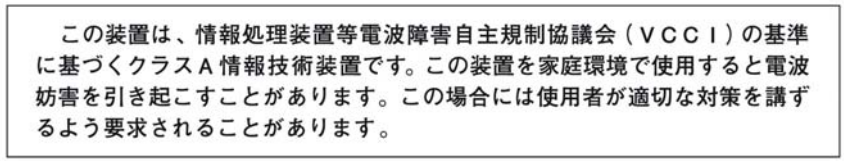
Note that Canadian Department of Communications (DOC) regulations provide, that changes or modifications not expressly approved by Malvern Instruments Limited could void your authority to operate this equipment.

This Class A digital apparatus complies with Canadian ICES-003.

Cet appareil numérique de la classe A est conforme à la norme NMB-003 du Canada.

VCCI acceptance (Japan only)

The Voluntary Control Council for Interference (VCCI) mark on this product signifies compliance to Japanese EMC regulations as specified by VCCI.



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Translation:
This is a Class A product based on the standard of the Voluntary Control Council for Interference by Information Technology Equipment (VCCI). If this equipment is used in a domestic environment, radio disturbance may occur, in which case the user may be required to take corrective actions.

Index

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